ADSORPTIVE REMOVAL OF TEXTILE DYES FROM AQUEOUS SOLUTION BY COCONUT COIR CARBON

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by

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CERTIFICATION OF APPROVAL

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Approved by,

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JANUARY 2009

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

NURUL AFIFAH BT MOHD ZAID

ABSTRACT

Activated carbon prepared from coconut coir was investigated in terms of its efficiency to adsorb textile dyes from aqueous solution and compared with that of a commercial charcoal activated carbon. The adsorbates used in this study were acid dyes, Acid Red and Acid Orange 7, which are commonly used in textile dyeing. The characteristics of the prepared coconut coir activated carbon such as pH and ash content were analyzed. Parameters such as initial dye concentration and contact time, pH and carbon dose that affect adsorptive removal of dye was studied in batch adsorption study. From the batch studies conducted, higher adsorption was seen for lower concentration of dyes and percentage removal increased with increasing contact time for both the coconut coir carbon and a commercial activated carbon. Maximum color removal was observed at pH 3. As the carbon dose increased, the percentage of dye removal also increased. Adsorption isotherm data fitted well to Freundlich and Langmuir isotherm models. According to the Freundlich isotherm, coconut coir activated carbon has higher adsorption capacity and lower adsorption intensity for the acid dyes compared to the charcoal activated carbon. According to Langmuir isotherm, the coconut coir activated carbon has appreciable adsorption capacity but lower adsorption energy for the acid dyes compared to the charcoal activated carbon.

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LIST OF ABBREVIATION AND NOMENCLATURES

ABBREVIATION

PPE	Personal Protective Equipment
US	United State

NOMENCLATURES

CO_2	Carbon Dioxide
HCl	Acid Hydrochloric
NaOH	Sodium Hydroxide
PAC	Powder Activated Carbon
GAC	Granular Activated Carbon
AC	Activated Carbon

CHAPTER 1

INTRODUCTION

1.1. Background Studies

Dye can be generally described as the coloring agent that is widely used in industries for colored substance such as textiles, food, paper, and carpet, rubber, cosmetic and plastic (Hoda *et al.*, 2005). There are many types of dye used in the industry such as reactive dyes, vat dyes, direct dyes, acid dyes, basic dyes and pigment dyes. The widespread used of these dyes has lead to concern over human health and environment as of their characteristics which are carcinogenic and mutagenic (Thinakaran *et al.*, 2008). The color removal from the effluent water is becoming an important objective of wastewater treatment processes. This goal is pursued not only because the legislation requirement has become more stringent but also if the water quality for recycle purposes is taken into account for the discharge of industrial waste into wastewater. Various chemical and physical methods have been proposed for the removal of dye from the effluent water. These techniques are nanofiltration, electrokinetics coagulation, liquid-liquid extraction, ozonation, biological process and adsorption (Hoda *et al.*, 2005; Xue *et al.*, 2008). Among these, adsorption has been found out to be the superior compared to the others techniques. It is due to its capability for efficiently adsorbing a broad range of adsorbates and its simplicity of design. The use of activated carbon as the adsorbent, however, is still considered expensive. Therefore, a study on the activated carbon prepared from agricultural waste, which is the coconut coir, is appropriate because it is available in large quantities (Santhy and Selvapathy, 2006; Yamin *et al.*, 2007).

1.2. Problem Statement

As a result of the dye production and also as a consequence of the dye usage in the textile industries, the issue of effluents discharge arises. The released dye into the effluent water or direct discharge of dyes into the municipal wastewater treatment plant and the environment will cause many problems to human being due health issues caused by the formation of toxic carcinogenic breakdown products (Thinakaran *et al.*, 2008). In the wastewater treatment, especially dye coloring industries need to include the process of color removal from the effluent water. This process requires chemical and physical methods such as liquid-liquid extraction, biological process and so on, which are uneconomical because of high operational cost and complicated design. For a long time treatment of effluents have concentrated mainly on two aspects which are regeneration of concentrated effluent with regards to saving of chemicals and lowering of chemical costs and treatment of effluent with high toxicity. Adsorption method has been implemented to remove dyes from effluent water (Thinakaran *et al.*, 2008; Saiful Azhar *et al.*, 2005). Commercial activated carbon has been successfully competitive and effective for adsorption process for removal of textile dye. However, the use of commercial activated carbon is not suitable due to high costs associated with production and regeneration of spent carbon (Erdem *et al.*, 2004). Thus, economical, effective and viable alternative adsorbent will be studied to replace the commercial activated carbon and be more applicable for low cost adsorption process (Saiful Azhar *et al.*, 2005).

1.3. Objectives and Scope of Study

The main objectives of the study were to remove the textile dyes, Acid Red and Acid Orange 7 from aqueous solution by a sorbent prepared from agricultural waste which is coconut coir in form of powdered activated carbon (PAC) and also to investigate the adsorption capacities and the effectiveness of the coconut coir activated carbon compared to a commercial activated carbon, charcoal activated carbon.

The aim of the present work was to investigate the removal of two dyes from acid class which is Acid Orange 7 and Acid Red from aqueous solution by adsorption

onto coconut coir carbon. Two tests of batch studies and adsorption isotherm studies were conducted. In batch studies, effect of adsorbent dosage, initial dye concentration, contact time and dye solution pH on adsorption by the activated carbon prepared from coconut coir were investigated. In the adsorption isotherm studies, isotherm data were treated according to Freundlich and Langmuir models. The fits of experimental data to these equations were examined.

1.4. Relevancy of the Project

Adsorbent obtained from agricultural waste, known as natural sorbent will be used instead of being throwing away and increase the waste in country. The recycling opportunities of this waste can be develop. Furthermore, as adsorbent for adsorption, coconut coir can replace the expensive commercial activated carbon in the market as the adsorbent is easily available and not bulky to handle. Low in density and high volatile content in coconut coir made it suitable to produce reasonably high quality and low density activated carbon.

1.5. Feasibility of the Project

The feasibility study of the project within the scope and time frame is conducted to get the best way of how to manage the entire task in completing the research project. For this first part of the research project, understanding of research previous done will be made. All relevant information will be gathered to be the guide in completing this study.

Preparation of adsorbent is the most crucial part to produce high quality of activated carbon. Preparation of activated coir carbon and preparation of dyes solution will also be preceded depending on the availability of equipment and workstation. Proper planning and procedures are developed to ensure a smooth project flow as well as to accomplish the project's objectives within the time period. The timeline of the study is shown in the Figure 3.11, Gantt chart.

CHAPTER 2

LITERATURE REVIEW

2.1. Adsorption

2.1.1. Adsorption Theory

Adsorption is the assimilation of a gas, liquid or dissolved substance by the surface of a solid and is a physicochemical treatment process of wastewater that is gaining prominence as a means of producing high quality effluent that is low in dissolved organic compounds. Conventional methods for the removal of dyes from wastewater include adsorption onto solid substrates, chemical coagulation, disinfection, and filtration and UV treatment. Adsorption has become a well-established separation technique to remove dilute pollutants as well as offering the potential for regeneration, recovery and recycling of the adsorbed materials (Noroozi *et al.*, 2008). The adsorption process is different from absorption process. Adsorption is the process by which molecules of a substance, such as a gas or a liquid, collect on the surface of another substance, such as a solid. Meanwhile, absorption is the processes by which the molecules are attracted to the surface but do not enter the solid's minute spaces (Atkins, 1994).

2.1.2. Adsorption Principles

Adsorption techniques for wastewater treatment have become more popular in recent years owing to their efficiency in the removal of pollutants too stable for biological methods. Adsorption can produce high quality water while also being a process that is economically feasible (Choy *et al.*, 2004).

Ruthven (1984) reported the chemical adsorption and physical adsorption. Chemical adsorption occurs when strong interparticle bonds are present between the adsorbate and adsorbent due to an exchange of electrons. Examples of such bonds are covalent and ionic bonds. Chemisorption is deemed to be irreversible in the majority of cases.

Physical adsorption occurs when weak interparticle bonds exist between the adsorbate and adsorbent. Examples of such bonds are van der Waals, Hydrogen and dipole-dipole. Physical adsorption, or van der Waals adsorption, usually occurs between the adsorbed molecules and the solid internal pore surface and is readily reversible (Christie, 2003).

Meanwhile, Suzuki (1990) covered the role of adsorption in water environmental processes and also covered the development of newer adsorbents to modernize the treatment systems and the role modeling of the findings plays in their development. Most adsorbents are highly porous materials. As the pores are generally very small, the internal surface area is orders of magnitude greater than the external area.

2.1.3. Adsorption Process

Adsorption is recognized as a significant phenomenon in most natural physical, biological and chemical processes. Sorption on solids, particularly active carbon, has become a widely used option for purification of water and wastewaters. It plays an important role in many other process of water treatment (Weber, 1972).

Metcalf and Eddy (2004) also mentioned the four definable steps of adsorption process: "Bulk solution transport involves the movement of the organic material to be adsorbed through the bulk liquid to the boundary layer of fixed film of liquid surrounding the adsorbent. Film diffusion transport involves the transport by diffusion of the organic material through the stagnant liquid film to the entrance of the pores of the adsorbent. Pore transport involves the transport of the material to be adsorbed through the pores by a combination of molecular diffusion through the pore liquid and/or by diffusion along the surface of the adsorbent. Adsorption involves the attachment of the material to be adsorbed to adsorbent at an available adsorption site.

The overall adsorption process consists of a series of steps in series. When the fluid is flowing past the particle in a fixed bed, the solute first diffuses from the bulk fluid to the gross exterior surface of the particle. Then the solute diffuses inside the pore to the surface of the pore. Finally, the solute is adsorbed on the surface. Hence, the overall

adsorption process is a series of steps (Christie, 2003). Table 2.1 below shows several reported method for dye removal. Each technology has its own advantages and drawback (Laszlo, 1994).

Table 2.1: Technologies use in dye removal

Principal existing and emerging processes for dyes removal

	Technology	Advantages	Disadvantages
Conventional treatment processes	Coagulation Flocculation	Simple, economically feasible	High sludge production, handling and disposal problems
	Biodegradation	Economically attractive, publicly acceptable treatment	Slow process, necessary to create an optimal favorable environment, maintenance and nutrition requirements
	Adsorption on activated carbons	The most effective adsorbent, great, capacity, produce a high quality treated effluent	Ineffective against disperse and vat dyes, the regeneration is expensive and results in loss of the adsorbent, non-destructive process
Established recovery processes	Membrane separations	Removes all dye types, produce a high-quality treated effluent	High pressures, expensive, incapable of treating large volumes
	Ion-exchange	No loss of sorbent on regeneration, effective	Economic constraints, not effective for disperse dyes
	Oxidation	Rapid and efficient process	High energy cost, chemicals required

(Source: Laszlo, 1994)

2.1.4. Adsorption Solution-Solid

Adsorption is a separation process where one or more components of a gas or liquid stream adsorbed on the surface of a solid adsorbent. The fluid is passed through the adsorbent and the solid particles adsorb the components from the fluid. Adsorption method has been used to remove dyes from effluent water (Allen and Koumanova, 2005; Christie, 2003).

In adsorption processes one or more components of a liquid stream are adsorbed on the surface of a solid adsorbent and a separation process is accomplished. In commercial processes, the adsorbent is usually in the form of small particles in a fixed bed. The fluid is passed through the bed and the solid particles adsorb components from the fluid. When the bed is almost saturated, the flow in this bed is stopped and the bed is regenerated thermally or by other methods so that desorption occurs. The adsorbed

material (adsorbate) is thereby recovered and the solid adsorbent is ready for another cycle of adsorption (Christie, 2003).

Positive adsorption in a solid-liquid system results in the removal of solutes from solution and their concentration at the surface of the solid, to such time as the concentration of the solute remaining in solution is in a dynamic equilibrium with that at the surface. At this position of equilibrium, there is a defined distribution of solute between the liquid and solid phases (Weber, 1972).

2.1.5. Adsorption by Activated Carbon

Adsorption is the process of accumulating substances that are in solution on a suitable interface. It also can be defined as a mass transfer operation in that a constituent in the liquid phase is transferred to the solid phase. The adsorption process has not been previously used extensively in wastewater treatment, but demands for a better quality of treated wastewater effluent, including toxicity reduction, have led to an extensive examination on use of the process of adsorption on activated carbon (Metcalf and Eddy, 2004).

The simplest way to conceptualize carbon adsorption is to think of the carbon particle as a porous ball. The water to be purified flows over the surface of, and between the balls. Solutes diffuse from the water stream into the pores of the ball and subsequently become adsorbed, or attached, to the surface of the pore.

Consequently, the rate of solute adsorption depends on how quickly a contaminant molecule can move from the bulk water flow to an unoccupied site within the balls' porous structures. The rate of adsorption is influenced by the number and size of the pores and temperature, but not the velocity with which the water flows over and between the balls.

The ideal particle would be small with many pores in order to minimize the distance the solute has to travel from the bulk fluid to an adsorption site. However, very small

particles are difficult to contain and cause large pressure drops across the carbon bed. Solutes move more slowly at low temperatures, so that the rate of adsorption decreases as the temperature of the water decreases.

As the water enters a fresh carbon bed, contaminating solutes are swept to the surface of the initial layer of porous balls. Some of the solutes find their way into the porous structure and are adsorbed. The partially purified water flows to succeeding layers, where the process is repeated, and on through the bed.

As the porous balls at the inlet of the bed become saturated with solute, the incoming water will not be purified until it encounters a subsequent layer, where the porous balls still have adsorptive capacity. With the flow rates normally used through carbon beds, solutes are delivered to the surface of the balls much more quickly than they can be adsorbed.

As a result, some solutes may appear in the product stream, although at reduced concentrations, even though the porous balls may still have capacity to adsorb more solute.

Activated carbon is the most popular adsorbent used in wastewater treatment and in the removal of dyes. Several equilibrium studies on the adsorption of dyes using activated carbon and its derivatives have been carried out. Some authors have investigated multi-component dye systems based on activated carbon and peat. Over the last decade, the low cost and commercial availability of biosorbents have stimulated great attention into the possibility of using various low cost adsorbents (Noroozi *et al.*, 2008).

2.1.6. Adsorption Isotherm

Equilibrium adsorption isotherms play an important role in the predictive modeling that is used for the analysis and design of adsorption systems. As such models can be used to predict the performance of an adsorption process under a range of operating conditions,

adsorption isotherms are invaluable tools for theoretical evaluation. However, no single model has been found to be generally applicable because although one isotherm equation may fit experimental data accurately under one set of conditions, it may fail entirely under another. Although considerable information has been collected for the adsorption of single component dyes by various adsorbents, many industrial situations involve the discharge of effluents that contain a mixture of several dyes; limited data is available on multi-component dye adsorption. However, an efficient, accurate and cost effective design should account for multi-component adsorption. Prediction of the equilibrium adsorption of mixtures from pure-component isotherms is important for practical application (Noroozi *et al.*, 2008).

Two most common isotherms which are used to describe experimental isotherm data are Freundlich and Langmuir isotherm. Freundlich isotherm is the most commonly used isotherm to describe the adsorption characteristic of activated carbon used in water and wastewater treatment (Tchobanoglous *et al.*, 2004). The Freundlich isotherm model takes the multilayer and heterogeneous adsorption into account (Hoda *et al.*, 2005). The Freundlich equation has the general form (Weber, 1972):

$$\frac{x}{m} = K_f C_e^{1/n}$$

The Freundlich equation is basically empirical but is often useful as a means for data description. Data are usually fitted to the logarithmic form of the equation (Weber, 1972):

$$\log\left(\frac{x}{m}\right) = \log K_f + \frac{1}{n}\log C_e$$

where: $\frac{x}{m}$ = amount of the adsorbate adsorbed per unit weight of adsorbent, mg adsorbent per g of activated carbon (mg/g)

K_f = adsorption capacity of activated carbon, (mg adsorbate/g activated carbon) (L water/ mg adsorbate) $^{1/n}$

C_e = concentration of the dye solution at equilibrium, mg/L

$1/n$ = adsorption intensity of activated carbon

When log *(x/m)* is plotted against *log* c_e and the data are treated by linear regression analysis, *1/n* and *K* constants are determined from the slope and intercept (Hoda *et al.*, 2005).

The Langmuir adsorption model is valid for single-layer adsorption. The Langmuir equation can be used for providing parameters (*a* and *b*) with which to quantitatively compare adsorption behavior in different adsorbate-adsorbent systems, or for varied conditions within any given system (Weber, 1972). The monolayer capacity can be represented by the expression:

$$\frac{x}{m} = \frac{abC_e}{1+C_e}$$

The linear form of the above equation is represented as:

$$\frac{C_e}{\left(x/m\right)} = \frac{1}{ab} + \frac{1}{a}C_e$$

where: a = the maximum adsorption capacity

b = the constant related to the free energy of adsorption

The values of *a* and *b* are calculated from the slope and intercept of the linear plot of $C_e/(x/m)$ versus C_e (Thinakaran *et al.*, 2008). The Langmuir isotherm is based on the assumption that adsorption takes place at specific homogeneous sites within the adsorbent and once a dye molecule occupies a site, no further adsorption takes place at the site (Langmuir, 1918).

2.2. Adsorbent (Activated Carbon)

2.2.1. Definition

The adsorbent is the solid, liquid, or gas phase onto which the adsorbate accumulates. The principal types of adsorbents include activated carbon, synthetic polymeric and silica-based adsorbent, although these adsorbents are seldom used for wastewater adsorption because of their high cost. The nature of activated carbon, the use of granular

carbon and powdered carbon has been discussed (Metcalf and Eddy, 2004). Adsorbents are materials with capability of adsorption and having the tendency to adsorb. Some of the best known and classic adsorbents are silica, alumina, and activated charcoal.

2.2.2. Activated Carbon Principles

Activated carbon is prepared by the pyrolysis of a variety of organic materials (for example, coconut shells, bones, coal, lignite, peat, petroleum, and wood) in closely regulated atmospheres. The porosity of activated carbon and, thus, its internal surface area, varies with the type of material used and the conditions of pyrolysis. The presence of oxygen alters the surface of the carbon inside the porous structure, changing its adsorptive properties.

Depending on the material from which the carbon is prepared, various amounts and types of residual metallic contaminants, including aluminum, may be left in the activated carbon. These contaminants have the potential to leach into water as it flows through the carbon.

Adsorbents combine chemical and physical processes to remove organic contaminants and the compounds that impart color, taste, and odor to water. The most commonly-used adsorbent is activated carbon—a substance which is quite similar to common charcoal. Activated carbon, however, is treated by heat and oxidation so that it becomes porous and able to readily adsorb, or capture, the impurities found in water.

Activated carbon also attracts not only known contaminants, but also naturally dissolved organic matter (much of which is harmless). Therefore, monitoring is needed to ensure that carbon doses are high enough to adsorb all contaminants. There are two different forms of activated carbon in common use, granular activated carbon (GAC) and powdered activated carbon (PAC). Physically, the two differ as their names suggest—by particle size and diameter. Overall, activated carbon is better than ion exchange for removing organic substances.

2.2.3. Application in Industries

Activated carbon is used in gas purification, gold purification, metal extraction, water purification, medicine, sewage treatment, air filters in gas masks and filter masks, filters in compressed air and many other applications. Carbon adsorption is used in drinking water to remove a wide variety of organic contaminants. It is not practical to attempt to list the wide range of organic chemicals versus the effectiveness of various types of carbon filter materials/products due to the wide variation in testing scenarios, concentrations of challenge materials, "contact time", flow rates and other else.

Suffice it to say that carbon possesses a variety of effectiveness while exposed to these organic compounds. A wide variety of industrial applications use coal-based carbon. Since it is not used for drinking water in this application, the levels of arsenic and other minerals in the carbon do not pose a health hazard.

Even coconut carbon filters will exhibit low levels of arsenic, probably through absorption of contaminated ground water. Rinsing or thoroughly flushing the carbon filter prior to use for drinking water purpose will substantially reduce the level of arsenic which may reside in loose carbon "fines" and thus be released in the drinking water during normal flow conditions. There are convenient Coir Bricks already packages as shown in Figure 2.1 below.

Figure 2.1: Coconut coir bricks

The grow coir is a natural fiber made from coconut husks. Sometimes the coir is called Coco Peat. However, the coconut coir is already known as a medium for hydroponics, container plant growing or outdoor gardening because it provides an excellent growing medium. Therefore, this experiment will be recovering other benefit of coconut coir as activated carbon for dye removal.

2.2.4. Physical Properties

Many adsorbents have been developed for a wide range of separations. Typically, the adsorbents are in the form of small pellets, beads, or granules ranging from about 0.1 mm to 12 mm in size, with the larger particles being used in packed beds. A particle of adsorbent has a very porous structure, with many fine pores and pore volumes up to 50% of total particle volume. The adsorption often occurs as a monolayer on the surface of the fine pores, although several layers sometimes occur (Christie, 2003).

Coir is a natural fiber derived from the husk of the coconut. The husk contains fiber and fine materials that constitute coco peat. Coir fiber is a homogenous material comprised of millions of capillary micro-sponges resulting in high water absorption and retention capacity. The fiber surface is covered with protrusions and small voids.

The important characteristics of an adsorbent are described in terms of both adsorptive capacity and physical properties (Noroozi *et al.*, 2008). The two differently processed coconut coir dusts from Mexico and Sri Lanka did not significantly differ in the microstructural parameters studied, whereas they differed remarkably in their particle size and therefore in their physical properties. Pithy tissue was the major constituent of coir dust. Coarse pithy tissue particles (larger than 1 mm) were highly porous, with an internal porosity of 41.0% by volume. These particles exhibited round-shaped external pores which ranged in diameter from 30 to 80 μm, with an average of 44.3 μm, and which accounted for a relative surface porosity of about 40.9%. The retting process (soaking in water) applied to the coconut husks in Sri Lanka partially degraded the pithy tissue and coir fibres. Coir dust from Mexico showed lower water-holding capacity and higher aeration than peat, whereas air–water relationships in coir dust from Sri Lanka were similar to those in peat. Although differences observed in microstructure and porosity characteristics could explain the differential physical properties, the major reason was the different size distribution of their constituent particles (Fornes *et al.*, 2002).

2.2.5. Classification

Adsorbent like activated carbon can be classified into two different type; powdered activated carbon (PAC), which has a diameter of less than 0.074mm, and granular activated carbon (GAC), which has a diameter greater than 0.1mm. The powdered activated carbon can be prepared by acid process. The powdered coconut coir activated carbon (CAC) which obtained is treated with emulsion of readily available synthetic polymer-polyvinyl acetate. Three parts by weight of powdered CAC is mixed with one part by weight of polyvinyl acetate to form a semisolid mass. Table 2.2 below shows the comparison of granular and powdered activated carbon. However, the source of the material used for the production of adsorbent will be affected on the every parameter mentioned in the table.

Table 2.2: Comparison of granular and powdered activated carbon

Parameter	Unit	Type of activated carbon[a]	
		GAC	PAC
Total surface area	m^2/g	700-1300	800-1800
Bulk density	Kg/m^3	400-500	360-740
Particle density, wetted in water	Kg/L	1.0-1.5	1.3-1.4
Particle size range	mm (μm)	0.1-2.36	(5-50)
Effective size	mm	0.6-0.9	na
Uniformity coefficient	UC	≤1.9	na
Mean pore radius	Å	16-30	20-40
Iodine number		600-1100	800-1200
Abrasion number	Minimum	75-85	70-80
Ash	%	≤8	≤6
Moisture as packed	%	2-8	3-10

(Source: Tchobanoglous *et al.*, 2004)

2.2.6. Manufacturing of Activated Carbon

Absorbent obtained from agricultural waste, known as natural sorbent will be used instead of throwing away and increase the waste in country. The recycling opportunities of this waste can be develop. Furthermore, the other alternative as absorbent for adsorption such as coconut coir can replace the expensive commercial activated carbon in the market, because the absorbent is easily available and not bulky to handle.

Nowadays, the color removal from the effluent water, especially from textile industries has been one of the concerns during the wastewater treatment process. To overcome the adverse effect caused from the releasing of the dye waste, activated carbon prepared from coconut coir is studied by investigating its efficiency to adsorb textile dyes from aqueous solution.

There are many efficient sorbent from agricultural waste and natural sorbent such as shell (Yamin *et al.*, 2007), treated sugarcane baggasse (Saiful Azhar *et al.*, 2005), sunflower seed hull (Thinakaran *et al.*, 2008), seed shells (Thinakaran *et al.*, 2008) and coir pith (Santhy and Selvapathy, 2006) have been used. Still, colored wastewater treatment needs new adsorbents that are economical, easily available and effective. Before using to remove the dye from aqueous solution, activated carbon must be prepared. Figure 2.2 shows the coconut coir that used in the study.

Figure 2.2: Coconut coir

2.2.7. Commercial Activated Carbon

Activated charcoal is a form of carbon that has been processed to make it extremely porous and thus have a very large surface area available for adsorption and chemical reactions. Due to its high degree of micro-porosity, just one gram of activated carbon has a surface area of approximately 500 m^2. Sufficiently activation for useful applications may come solely from the high surface area, though further chemical treatment often enhances the adsorbing properties of the material.

2.3. Adsorbate (Dye)

2.3.1. Definition

Adsorbate is a substance that is adsorbed. Adsorbate is a material that has been or is capable of being adsorbed. The adsorbate is the substance that is being removed from the liquid phase at the interface (Metcalf and Eddy, 2004). Dyes are chemicals which on binding with a material will give color to the material. Dyes are ionic, aromatic organic compounds with structures including aryl rings which have delocalized electron systems. The color of a dye was provided by the presence of a chromophore group (Allen and Koumanova, 2005). There are many types of dyes used in the industry such as reactive dyes, vat dyes, direct dyes, acid dyes, basic dyes and pigment dyes, which are carcinogenic and mutagenic.

2.3.2. Acid Dye

Acid dye is a member of a class of dye that is applied from an acidic solution. In textiles, acid dyes are effective on protein fibers, i.e. animal hair fibers like wool, alpaca and mohair. They are also effective on silk and cotton as well as to synthetics like polyesters, acrylic and rayon. But they are not substantive to cellulosic fibers. They are also used in paints, inks, plastics and leather. Acid dyes are generally divided into three classes which depend on fastness requirements, level dyeing properties and economy. The

classes overlap and generally depend on type of fiber to be coloured and also the process used. Acid dyes are thought to fix to fibers by hydrogen bonding, Van der Waals forces and ionic bonding. The strength (fastness) of this bond is related to the desire/ chemistry of the dye to remain dissolved in water over fixation to the fiber. The chemistry of acid dyes is quite complex. Dyes are normally very large aromatic molecules consisting of many linked rings. Acid dyes usually have a sulphonyl or amino group on the molecule making them soluble in water. Water is the medium in which dyeing takes place.

Hoda *et al.* (2006) covers the acid dyes that related in basic structure of Anthraquinone type, Azo dye and Triphenylmethane. Many acid dyes are synthesized from chemical intermediates which form anthraquinone-like structures as their final state as shown in the Figure 2.3(a). Many blue dyes have this structure as their basic shape. The structure predominates in the leveling class of acid dye.

Acid dyes having structures related to triphenylmethane predominate in the milling class of dye. There are many yellow and green dyes commercially applied to fibers that are related to triphenylmethane. The structure is shown in Figure 2.3(b).

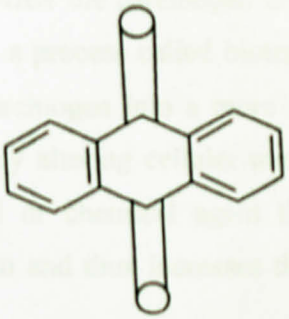


Figure 2.3(a): Anthraquinone Structure

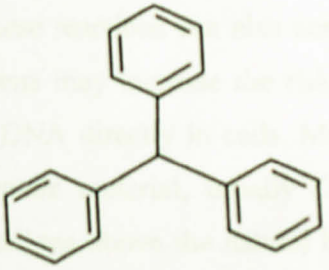


Figure 2.3(b): Triphenylmethane Structure

Meanwhile, the structure of azo dyes is based on azobenzene, Ph-N=N-Ph. Although Azo dyes are a separate class of dyesuff mainly used in the dyeing of cotton (cellulose) fibers many acid dyes have a similar structure, most are red in color.

2.3.3. Dye in the Environment

Waste aqueous effluent containing color compounds causes serious environmental problems. It has been estimated that about 9% of the total amount (450 000 tons) of dyestuffs produced in the world are discharged in textile wastewater. The greatest environmental concern with dyes is their absorption and reflection of sunlight entering the water which interferes with the growth of bacteria to levels insufficient to biologically degrade impurities in the water. Color in effluents can cause problems in several ways; dyes can have acute and/or chronic effects on exposed organisms depending on the exposure time and dye concentration; dyes are inherently highly visible meaning that concentrations as low as 0.005ppm capture the attention of both public and the authorities; the absorb and reflect sunlight entering water and so can interfere with the growth of bacteria and hinder photosynthesis in aquatic plants (Allen and Koumanova, 2005).

The effluent discharged from textile industries is highly colored and disposal of this colored water into receiving water body not only causes damage to aquatic life, but also to human beings, by producing carcinogenic and mutagenic effects (Thinakaran *et al.*, 2008). After the carcinogen enters the body, the body makes an attempt to eliminate it through a process called biotransformation. But these reactions can also convert a less toxic carcinogen into a more toxic one. Carcinogens may increase the risk of getting cancer by altering cellular metabolism damaging DNA directly in cells. Mutagen is a physical or chemical agent that changes the genetic material, usually DNA of an organism and thus increases the frequency of mutations above the natural background level.

Growing concern about environmental issues has prompted the textile industry to investigate appropriate and environmentally friendly treatment technologies.

2.3.4. Dye in Industries

Dyeing is the process of imparting colours to a textile material in loose fibre, yarn, cloth or garment by treatment with a dye. For most of the thousand years in which dyeing has been used by humans to decorate clothing and fabrics. The primary source of dye has been nature, with the dyes being extracted from animals or plants. In the last 150 years, man has produced artificial dyes to achieve a broader range of colors, and to render the dyes more stable to resist washing and general used. Differential classes of dye are used for different types of fiber and at different stages of the textile production process from loose fibers through yarn and cloth to made garments (Bhardway and Jain, 1996). Acrylic fibers are dyed with basic dyes, nylon and protein fibers such as wool and silk are dyed with acid dyes, polyester yarn is dyed with disperse dyes. Cotton is dyed with a range of dye types including vat dyes which are similar to the ancient natural dyes and modern synthetic reactive and direct dyes (Bhardway and Jain, 1996). Meanwhile, Thinakaran *et al.* (2008) study on the dyes which are acid dyes and identify that acid dyes are used by several industries, such as textile, paper, printing and plastics to color their product. Figure 2.4 is shown some of the acid dyes at market in India:

Figure 2.4: Pigment for sale at a market in Goa, India
(Source: Bhardway and Jain, 1996)

There are many textile industries operating in Malaysia such as producing of cotton, nylon, and so on. In Perak, there are several places that produce textile such as Kamunting Textile Industries Sdn. Bhd and Perak Textile Mill, Kuala Kangsar. Colored dye wastewater arises as a direct result of the production of the dye and also consequence of its use in the textile industries. There are more than 100 000

commercially available dyes with over $7x10^5$ tonnes of dyes produced annually (Robinson *et al.*, 2001).

2.4. Dye Removal by Activated Carbon

Removal of dyes from wastewater before discharging to environment and from raw water before offering it to public use is essential for the protection of health and environment (Hoda *et al.*, 2006). Textile Industries consume large volumes of water and chemicals. Wastewater streams from the textile dyeing operation contain unused dyes (about 8–20% of the total pollution load due to incomplete exhaustion of the dye) and auxiliary chemicals along with large amounts of water. The presence of dyes in water sources is aesthetically unacceptable and may be visible at concentration as low as 1 ppm (Noroozi *et al.*, 2008).

Activated carbon treatment of wastewater is usually thought of as a polishing process for water that has already received normal biological treatment. The carbon in this case is used to remove dye solution in the industry's effluent water (Metcalf and Eddy, 2004). Most of the commercial industries use activated carbon as adsorbent to remove color from wastewater (Tunali *et al.*, 2006). The reason for their adsorbing characteristics is their enormous surface area per unit weight. When the surface of the adsorbent is saturated by the adsorbate, a decrease in absorbance will be observed. This is due to the limited number of surface sites available for chemisorptions.

At the end of the study, the capacity of the dye removal by adsorption process of acid textile dyes namely, Acid Orange 7 and Acid Red onto coconut coir activated carbon will be investigated on the various experimental parameters such as effect of initial dye concentration and contact time, effect of pH solution and effect of carbon dosage.

CHAPTER 3

MATERIALS AND METHODS

3.1. Acid Dyes

The adsorbate Acid Red and Acid Orange 7 were obtained from Euro Chemo-Pharma Sdn. Bhd. (Penang, Malaysia). The dyes were chosen as an adsorbate because it is commonly used in dyeing of textiles and is a dye contaminant in discharged effluents.

Acid dye is a member of a class of dye that is applied from an acidic solution. Acid dyes usually have a sulphonyl or amino group on the molecule making them soluble in water. Water is the medium in which dyeing takes place.

Azo dyes are a separate class of dyestuff mainly used in the dyeing of cotton (cellulose) fibers many acid dyes have a similar structure, most are red in color. Figure 3.1 below shows an image of its molecular structure of Acid Red 266.

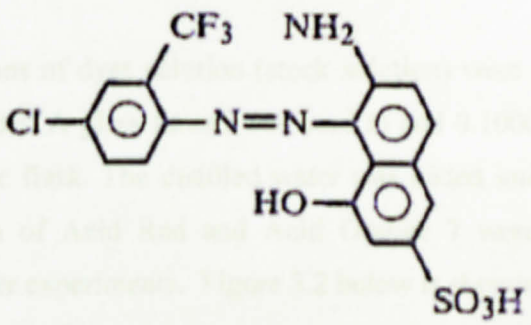
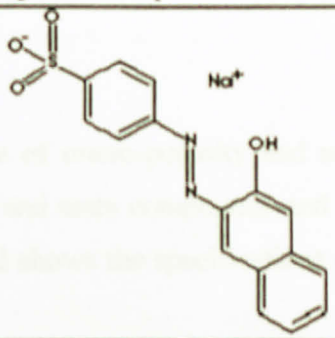


Figure 3.1: Molecular Structure of Acid Red 266

Meanwhile Table 3.1 shows the characteristics of Acid Orange.

Table 3.1: Characteristics of Acid Orange

ACID ORANGE	
CI Name	Acid Orange 7
Other Name	Acid Orange II
Physical State	Yellow powder (produce an orange-pink color)
Molecular Weight	350.32
Solubility	Water (116 g/L)
Chemical Name	4-(2-Hydroxy-1-naphthylazo)benzenesulfonic acid sodium salt; Sodium 4-[(2-hydroxy-1-naphthyl)azo]benzenesulphonate
Molecular Structure	

3.2. Dye Concentration

100 mg/L concentrations of dyes solution (stock solution) were prepared by weighting 0.1000 g of dyes powder. A glass funnel was used to add 0.1000 g of dye powder into 1000 mL of volumetric flask. The distilled water was added into the volumetric flask. This standard solution of Acid Red and Acid Orange 7 were diluted into specific concentration for further experiments. Figure 3.2 below is showing the stock solution of Acid Red and Acid Orange 7.

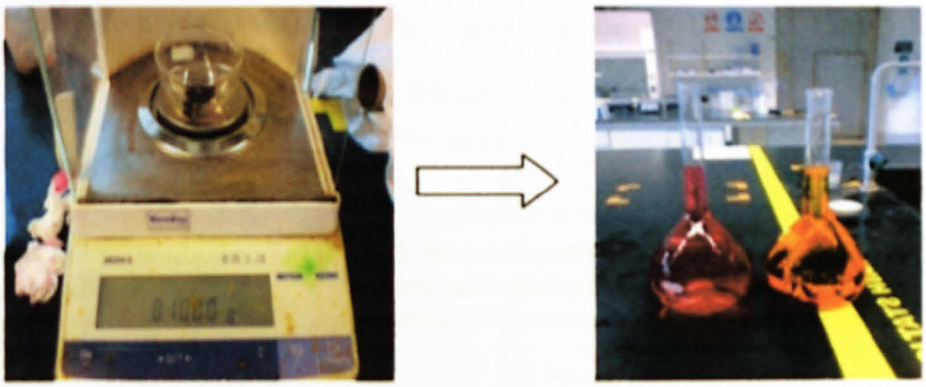


Figure 3.2: Dye Solution (Acid Red and Acid Orange 7)

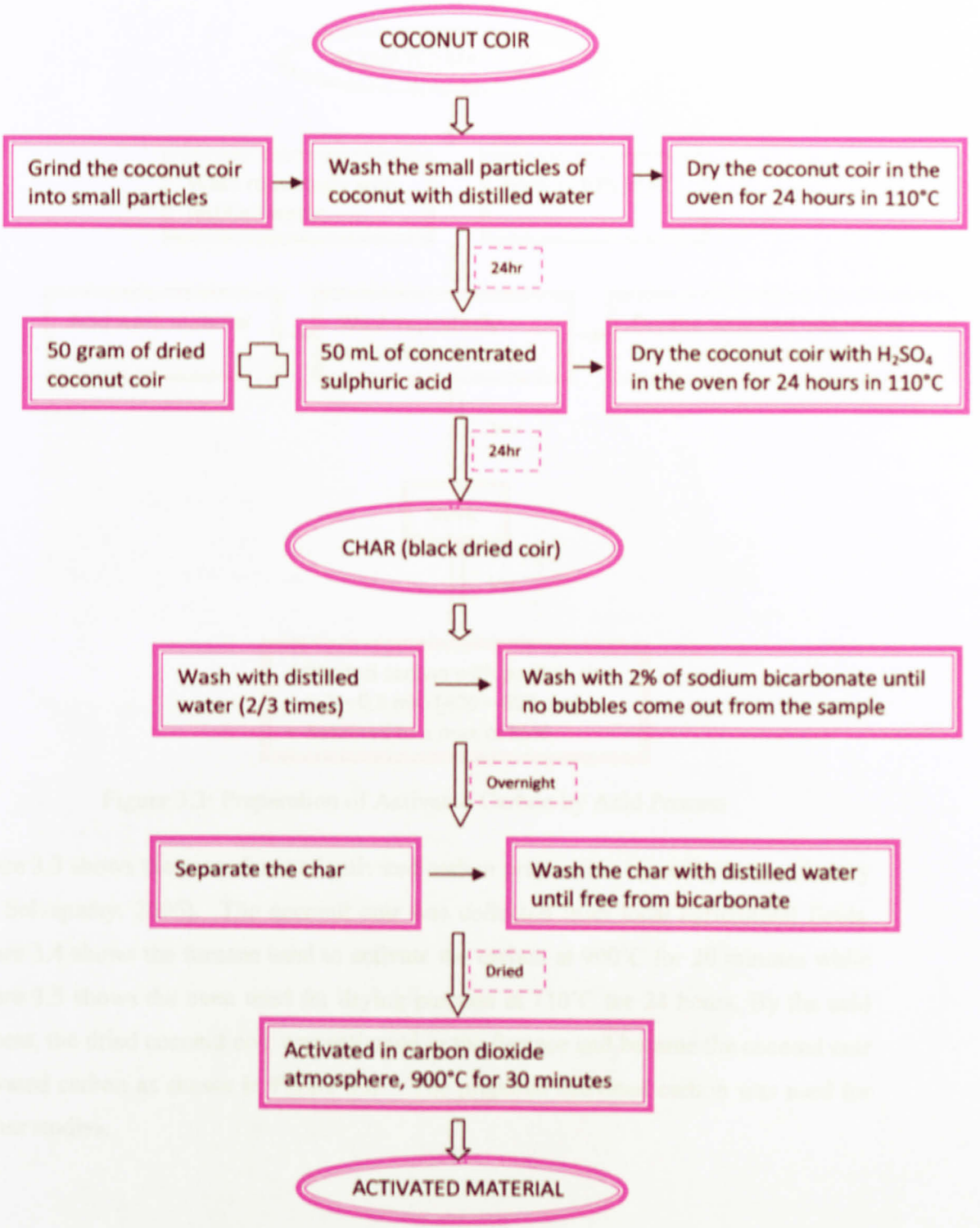
3.3. Commercial Activated Carbon

Charcoal activated carbon has high degree of micro-porosity and supplied by Benua Sains Sdn. Bhd. for the removal of odour and taste compounds and dissolved organic compounds from water treatment. Table 3.2 shows the specifications of the commercial charcoal activated carbon.

Table 3.2: Characteristic of charcoal activated carbon

CHARCOAL ACTIVATED CARBON	
Brand	Merck
Supplier	Benua Sains Sdn. Bhd., Puchong, Selangor
Molar Mass	12.01 g/mol
Density	2 g/cm^3
Effective Size	2.5 mm
pH Value	6 (Slurry)
Form	Granular (Solid)
Color	Black
Odour	Odourless
Bulk Density	400 kg/ m^3
Solubility	In water

3.4. Preparation of Coconut Coir Activated Carbon



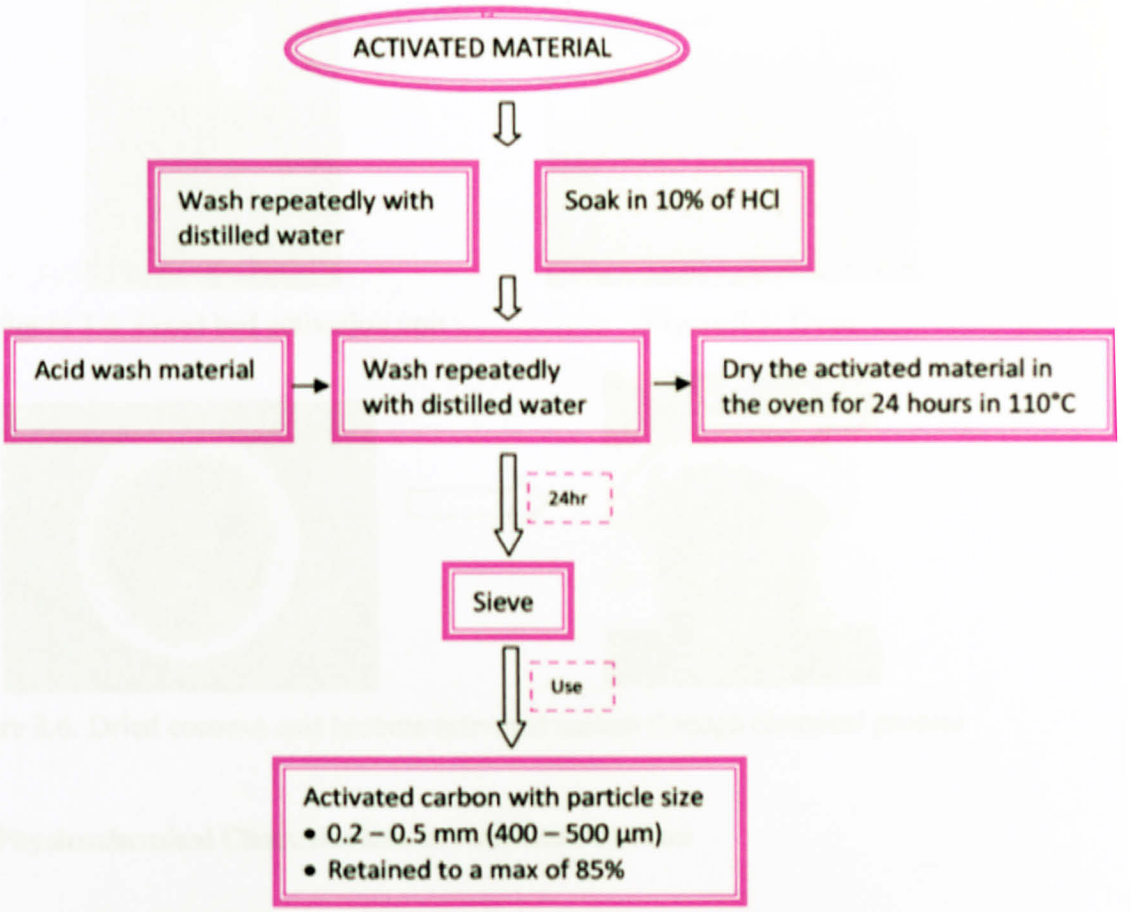


Figure 3.3: Preparation of Activated Carbon by Acid Process

Figure 3.3 shows the procedures of activated carbon preparation by acid process (Santhy and Selvapathy, 2006). The coconut coir was collected from local agricultural fields. Figure 3.4 shows the furnace used to activate the carbon at 900°C for 30 minutes while Figure 3.5 shows the oven used for drying purpose at 110°C for 24 hours. By the acid process, the dried coconut coir was activated in the furnace and became the coconut coir activated carbon as shown in Figure 3.6. The prepared activated carbon was used for further studies.

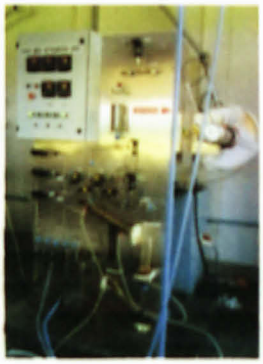


Figure 3.4: Fixed bed activation unit

Figure 3.5: Oven

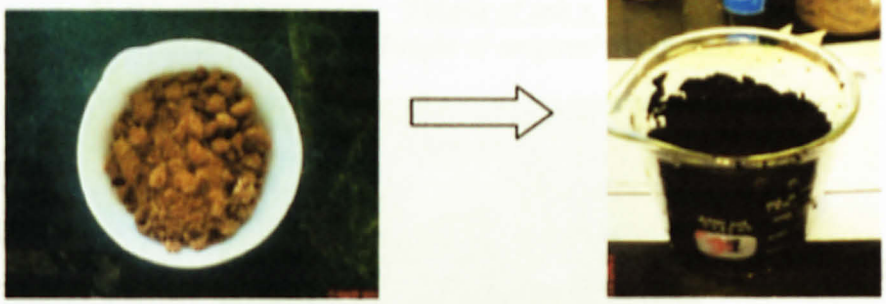


Figure 3.6: Dried coconut coir become activated carbon through chemical process

3.5. Physicochemical Characteristic of Activated Carbon

3.5.1. pH

1.0 g of coconut coir carbon sample was immersed and stirred in 100 ml of distilled water. After 1 hour, the pH of slurry was measured by pH meter shown in the Figure 3.7 and the pH of the carbon was determined (Bhardway and Jain, 1996). The same procedures were repeated 4 more times to get other 4 readings.

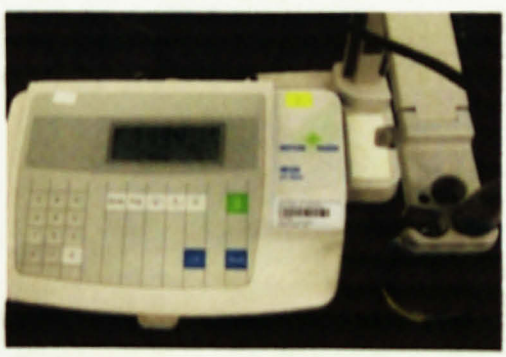


Figure 3.7: pH meter

3.5.2. Ash Content

The initial weight of the clean porcelain bowl and the initial weight of approximately 1 g of coconut coir carbon in clean porcelain bowl were recorded. The powdered carbon sample in porcelain bowl was burned in muffle furnace at 900°C for 12 hours. Figure 3.8 shows the equipment to investigate the ash content. The final weight of the remaining ash in the porcelain bowl was recorded (Saiful Azhar *et al.*, 2005). The percentage of ash content was calculated as the equation below (Santhy and Selvapathy, 2006):

$$\% \text{ ash content} = \frac{\text{Weight of ash} \times 100\%}{\text{Weight of original Sample}}$$

Figure 3.8: Muffle furnace

3.5.3. Scanning Electron Micrograph

Small amount of the coconut coir, coconut coir activated carbon and charcoal activated carbon were put on the clean sample plate. The samples were examined by using Leo 1430 VPSEM. The scanning electron micrograph was obtained and analyzed.

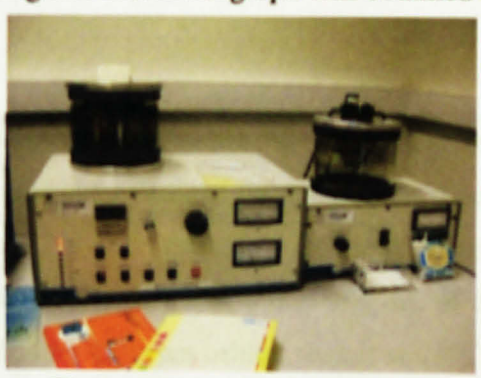


Figure 3.9: Leo 1430 VPSEM

3.6. Adsorption Study

3.6.1. Batch Study Using Coconut Coir Carbon

Batch experiments were carried out after the coconut coir activated carbon was obtained. The effect of various parameters such as initial concentration, contact time and adsorbent dose on the adsorption capacity was investigated.

3.6.1.1. Effect of initial concentration and contact time

Acid Red and Acid Orange 7 aqueous solutions were prepared. 100 mL of different initial concentration; 20 mg/L and 40 mg/L with pH 6.0 of every acid dye solutions were taken in a series of 125 mL conical flasks containing 0.5 g of powdered coconut coir carbon (400-500 μm particle size). The flasks were equilibrated by 150 rpm orbital shaker at 25°C for a pre-determined time. The progress of adsorption during the experiment was determined by taking the aliquots after desired contact time which is every 1 hour in range of 1 to 5 hours, filtering trough Whatman No.1 filter paper. The supernatant was analyzed spectrophotometrically for every dye concentration which is 20 mg/L and 40 mg/L. At time t=0 and equilibrium, the dye concentrations were measured using UV/VIS spectrophotometer (DR2800) where four vials filled with 10 mL of every concentration of dye solutions; Acid Red and Acid Orange 7 was taken. The absorbance of Acid Red at 505 nm and Acid Orange 7 at 485 nm was read and recorded (Senthilkumar *et al.*, 2006). The amount of dye adsorbed was calculated.

3.6.1.2. Effect of pH

Effect of pH was studies by varying the pH value. Concentrations of 40 mg/L of Acid Red and Acid Orange 7 aqueous solutions were prepared. The pH of the dye solutions were adjusted by using 0.02 N NaOH or 0.02 N HCl and varied from pH 3 to pH 11. 0.5 g of coconut coir activated carbon was added into 100 mL of 40 mg/L of dye solutions. The flasks were equilibrated by 150 rpm orbital shaker at 25°C for 2 hours. The reading

for every solution was taken after contact time 2 hour, filtering trough Whatman No.1 filter paper. The dyes with different pH were measured using UV/VIS spectrophotometer (DR2800) where vials filled with 10 mL of every dye solutions. The amount of dye adsorbed was calculated (Senthilkumar *et al.*, 2006).

3.6.1.3. Effect of Adsorbent Dose

Effect of adsorbent dose was studied by varying amount of powdered coconut coir activated carbon weight from 0.2 g to 1.0 g in 100 mL of 50 mg/L dye concentration for Acid Red and Acid Orange 7 (Senthilkumar *et al.*, 2006). Concentrations of 50 mg/L of Acid Red and Acid Orange 7 aqueous solutions were prepared. 100 mL of Acid Red and Acid Orange 7 at pH 3 was taken in a series of 125 mL conical flasks and 0.2 g, 0.4 g, 0.6 g, 0.8 g and 1.0 g of carbon were added into the flasks. The flasks were equilibrated by 150 rpm orbital shaker at 25°C for 2 hours. The aliquots were taken after 2 hours, filtering trough Whatman No.1 filter paper. The dyes with different adsorbent dose were measured using UV/VIS spectrophotometer (DR2800) where vials filled with 10 mL of every dye solutions. The amount of dye adsorbed was calculated.

3.6.2. Batch Study Using Commercial Activated Carbon

The same method was also conducted for the commercial activated carbon; charcoal carbon which was obtained from the manufacturer to compare its performance with the coconut coir activated carbon.

3.7. Adsorption Isotherm Study

Only when the result obtained from the batch study was favorable, the author will proceed with the adsorption isotherm studies. The concentration of both Acid Red and Acid Orange 7 were varied by ten different concentrations, which is from 10 mg/L to 100 mg/L. All the solutions were adjusted at pH 3 which is at the optimum condition. It was equilibrated for 2 hours after 0.5 g of coconut coir activated carbon was added inside 100 mL of the Acid Orange 7 solution and 0.6 g of coconut coir activated carbon was added inside 100 mL of the Acid Red solution. The flasks were equilibrated by 150

rpm orbital shaker at 25°C. The aliquots were taken after 2 hours, filtering trough Whatman No.1 filter paper. The dyes with different adsorbent dose were measured using UV/VIS spectrophotometer (DR2800) where vials filled with 10 mL of every dye solutions. The final concentration of the solutions was measured. The same experiment procedures were done using the commercial charcoal activated carbon.

The experimental data was analyzed. For the Freundlich Isotherm, the value of K (y-intercept) and $1/n$ (slope) which indicated the adsorption capacity and adsorption intensity of coconut coir carbon was compared to charcoal activated carbon. Meanwhile, for the Langmuir Isotherm, the value of a and b which indicating the adsorption capacity and adsorption energy of coconut coir carbon was compared to charcoal activated carbon. The calculation for all adsorption isotherms was attached in Appendix D and Appendix E.

Figure 3.10: Shaker Bottles on the Orbital Shaker

3.8. Data Collection and Schedule

All the data and information that are related to the project were obtained from experimental analysis and many references. Information from journal, website on related topic and text books were gathered to achieve the objective of the research which is to investigate the efficiency of coconut coir activated carbon. Meanwhile, the project schedule is shown on the Gantt chart.

PROJECT PHASE	week														
	1	2	3	4	5	6	7	8	9		10	11	12	13	14	15
Weekly Meeting with Supervisor	*	*	*	*	*	*	*	*	*							
Project Work																
• Preparation of activated carbon																
• Adsorption studies by coconut coir carbon / Batch study																
a) Effect of initial concentration and contact time																
b) Effect of carbon dose																
• Adsorption studies by existed coal carbon																
a) Effect of initial concentration and contact time																
• Physicochemical characteristics of the carbon																
a) SEM photograph																
b) PH measurement																
c) Ash content																
Submission of combined progress report																
Project Work																
• Adsorption studies by coconut coir carbon / Batch Study																
a) Effect of PH																
• Adsorption Isotherm																
a) Freundlich Isotherm																
b) Langmuir Isotherm																
Poster presentation																
Submission of Project Dissertation report																
Oral Presentation																

*Supervisor on Medical Leave

Figure 3.11: Gantt chart of the Project Schedule

3.9. Tools

- Equipment : Spectrophotometer DR2800, oven, tube furnace, sieve analysis, balance-weight, glassware (Figure 3.12), muffle furnace, Leo 1430 VPSEM, pH meter, fume cupboard and rotary mechanical shaker
- Chemical : Sulphuric acid, sodium bicarbonate, HCL, dye solutions; Acid Red and Acid Orange 7 and NaOH
- Material : Whatman No.1 filter paper
- Tool : Cutter, blender and grinder
- Software : Microsoft Office Excel and Microsoft Word

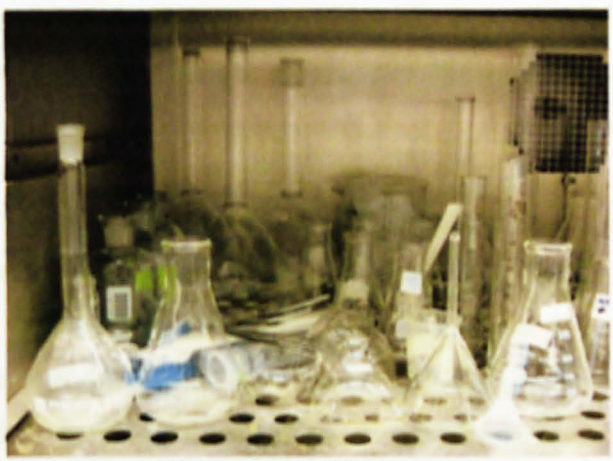


Figure 3.12: Glassware

3.10. Hazard Analysis

Hazard analysis is a systematic process and proactive approach for identifying hazards and recommending corrective actions. Hazard sources can be classified into sources of motion, sources of extreme temperatures, types of chemical exposures, sources of harmful dust, sources of light radiation, sources of sharp objects and any electrical hazards.

In this study, chemical laboratory will become a workplace to prepare the adsorbent, adsorbate and for the adsorption process. Working in hot conditions can cause health effects ranging from discomfort to serious illness. Therefore, identify the information on heat, temperature and pressure hazards at laboratory to overcome the hazards associated with the extreme heat, temperature and pressure (110°C - 900°C). In this study, hot usage of furnace, fixed bed activation unit and oven was identified. To overcome any hazard, wear the gloves when taking out the sample from oven can protect the heat from human body because of this mechanism of heat exchange can be altered dramatically. The operation must be under laboratory technician's supervision.

Second hazard is mechanical hazard, especially while using cutter, blender or grinder to grind the coconut coir. Body parts are in contact with sharp edges. This hazard will occur at the point of cutting operation. Safeguards are essential for the protection purposes and prevention from injuries. Gloves must be worn to avoid the sharp blade and the operation must be under laboratory technologist's supervision.

Chemical hazard exposures while handling the chemical in the chemical process of preparation the activated carbon and pH studies. Concentrated acid or alkali is corrosive and must therefore be handled with appropriate care, since it can cause skin burns, permanent eye damage and irritation. Latex gloves offer no protection, so rubber glove, should be worn when handling the compound. Certain chemicals are very high in concentrated, handle it inside laboratory fume hood and avoid smelling the fume of the chemicals. Figure 3.13 shows the symbol of corrosive chemicals.

Any dyes including acid dyes have the ability to affect humans due to their complex molecular structure and the way in which they are metabolized in the body. This is extremely rare nowadays as we have greater understanding through experience and knowledge of dyestuffs themselves. Some acid dyes are used to color food. We wear fabrics every day exposing our skin to dyes. The greatest risk of disease or injury due to dyes is by ingestion or exposure to dye dust. These scenarios are normally confined to textile workers. Whereby the dye itself is normally non toxic, the molecules are metabolized (usually in the liver) where they may be broken down to the original

intermediates used in manufacture. Thus many intermediate chemicals used in dye manufacture have been identified as toxic and their use restricted. There is a growing trend among governments to ban the importation of dyes synthesized from restricted intermediates. For example: the dye CI Acid red 128 is banned in Europe as it was found to metabolize in the body back to ortho-toluidine, one of its chemical intermediates. Many intermediates used in dye manufacture such as o-toluidine, benzidine etc. were found to be carcinogenic. All the major chemical companies have now ceased to market these dyes. Some, however, are still produced but they are found to be totally safe when on the fiber in its final state. The use of these dyes is declining rapidly as cheap and safer alternatives are now easily available. Thus, rubber gloves and mask must be worn when preparing the acid dye solution and also use spatula to take the chemical powder.

To prevent any accident happen and to ensure a safe work flow is practiced, student must attend the briefing session about laboratory in how to use or handle the tools, equipments or chemicals in proper ways. For the safety precaution, student must wearing appropriate personal protective equipment, PPE and clothing such as lab coat and closed shoe while entering the laboratory area. Prevent human contact with any potentially harmful machine part and handle properly the chemicals.

Figure 3.13: Symbol of corrosive chemical

CHAPTER 4

RESULTS AND DISCUSSION

4.1. Standard Curve for Acid Red and Acid Orange 7

For the result, the graphs of absorbance, A versus wavelength, nm for the dye concentration of 100 mg/L for Acid Red was obtained as the graph below. The values of absorbance and the wavelength were observed by varying the wavelengths for the dye concentration to obtain the maximum absorbance. From Figure 4.1, the standard wavelength for concentration of 100 mg/L of dye solutions can be determined by observing the maximum absorbance which is 505 nm for the Acid Red.

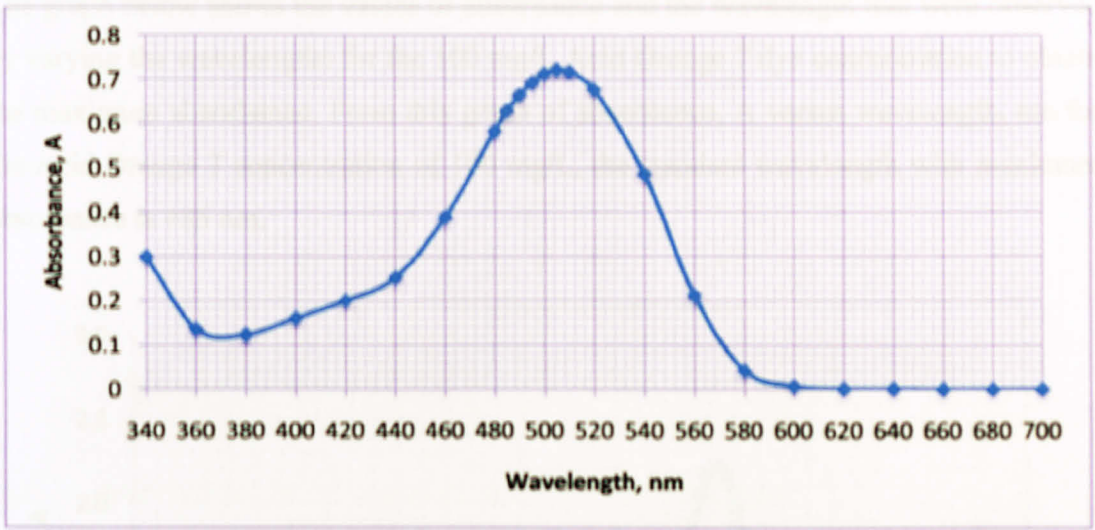


Figure 4.1: Absorbance versus Wavelength for Acid Red

Meanwhile for the graphs of absorbance, A versus dye concentration, mg/L were plotted to obtain the straight line of graph for Acid Red dye accordance to each standard wavelength. The standard curve was obtained by varying the dye solution concentration for dye as shown on the graph below, measurement of the absorbance; A on the spectrophotometer was done at the standard wavelength (maximum wavelength of 505 nm for the Acid Red). At the end, ensure to get a very high value of R^2 which is 0.997 for the Acid Red. The graph shows that the absorbance is directly proportional to the dye concentration.

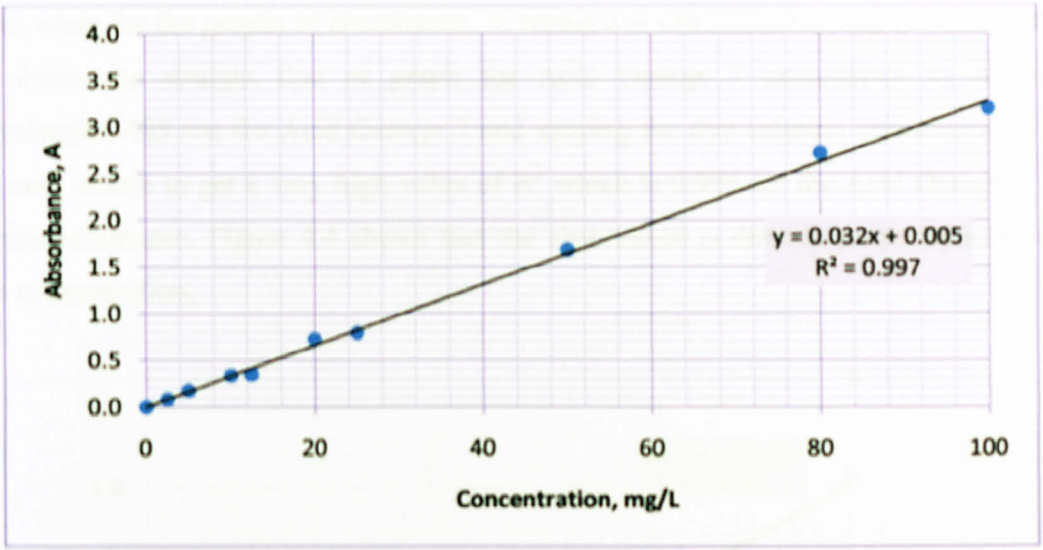


Figure 4.2: Standard Curve for Acid Red (Wavelength = 505 nm)

The graph below shows the values of absorbance and the wavelength that were observed by varying the wavelengths for the 100 mg/L Acid Orange 7 dye concentration to obtain the maximum absorbance. From this graph of absorbance, A versus wavelength, nm for the Acid Orange 7 concentration of 100 mg/L, the standard wavelength with maximum absorbance is 485 nm.

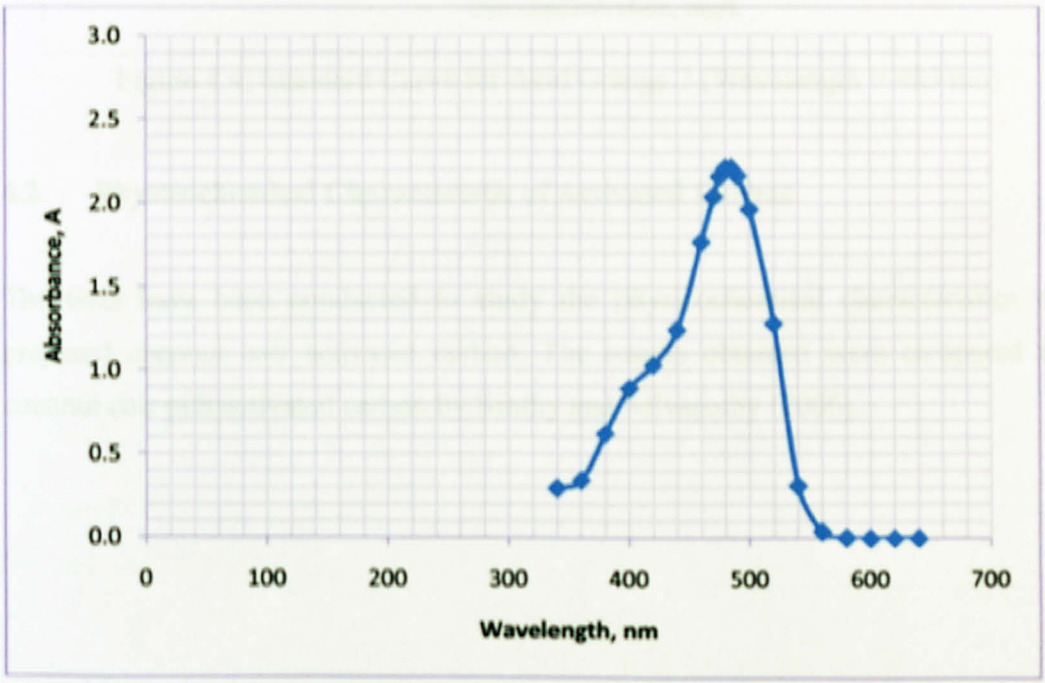


Figure 4.3: Absorbance versus Wavelength for Acid Orange 7

Meanwhile for the graphs of absorbance, A versus dye concentration, mg/L were plotted to obtain the straight line of graph for Acid Orange 7 accordance to standard wavelength, 485 nm for Acid Orange 7 and varying the dye solution concentration. At the end, ensure to get a very high value of R^2 which is 0.996 for the Acid Orange 7 for further references. Figure 4.4 shows that the absorbance is directly proportional to the dye concentration.

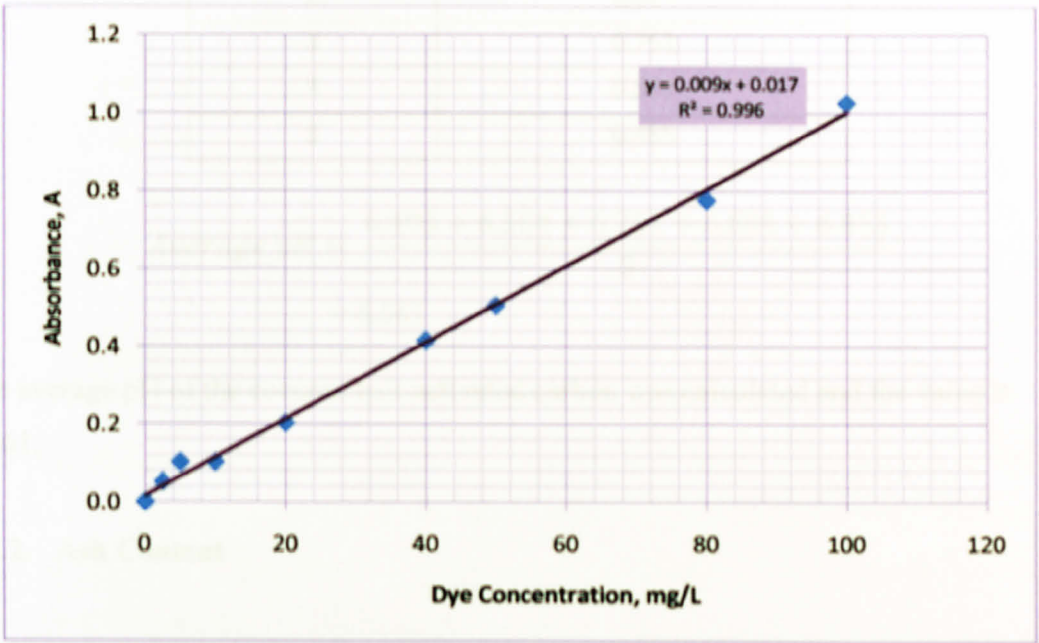


Figure 4.4: Standard Curve for Acid Orange 7 (Wavelength = 485 nm)

4.2. Physicochemical Characteristic of Activated Carbon

The tests have been conducted to study the physicochemical characteristics of the prepared coconut coir activated carbon. The results obtained were compared to the coconut coir pith activated carbon by Santhy and Selvapathy (2006).

4.2.1. pH

Table 4.1 below shows the readings of pH for different sample of coconut coir carbon.

Table 4.1: pH of the carbon

SAMPLE	pH
1	6.495
2	6.504
3	6.751
4	6.601
5	6.455

$$Average\ pH = \frac{6.495 + 6.504 + 6.751 + 6.601 + 6.455}{5}$$

$$= 6.561$$

The average pH of the coconut coir activated carbon was calculated and the value is 6.561.

4.2.2. Ash Content

Table 4.2 below summarized the ash content of prepared coconut coir carbon obtained in 125 mL size of porcelain bowl after burning in muffle furnace at 900°C.

Table 4.2: Ash content of the carbon

PARAMETER	MASS, g
Weight of the porcelain bowl	26.9433
Initial weight of the porcelain + sample	27.9433
Final weight of porcelain + sample	26.9752

The calculation to determine the ash content is shown below and the calculated percentage of the ash content is 3.19%:

$$\frac{26.9752 - 26.9433}{27.9433 - 26.9433} \times 100\% = 3.19\%$$

4.2.3. Comparison with Coconut Coir Pith Activated Carbon

The comparison of all the characterization tests between the prepared coconut coir activated carbon with the coconut coir pith activated carbon by Santhy and Selvapathy (2006) are presented in Table 4.3. The pH of both carbons can be observed from the table and show almost the same values. However, the coconut coir activated carbon has higher ash content compare to coconut pith activated carbon.

Table 4.3: Comparison of coconut coir carbon and coconut coir pith carbon

Characteristic	Coconut coir AC	Coconut coir pith AC
pH	6.56	6.50
Ash content, %	3.19	2.93

4.2.4. Scanning Electron Micrograph

Scanning electron micrograph (SEM) of coconut coir fiber, coconut coir activated carbon and charcoal activated carbon were observed by Variable Pressure Scanning Electron Microscope (VPSEM) with different magnifications. The figures below show the SEM of the samples.

4.2.4.1. Coconut Coir

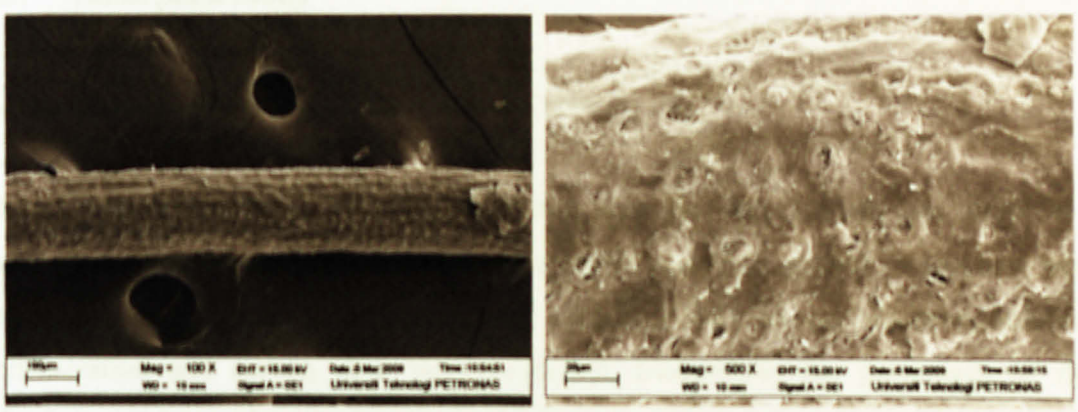


Figure 4.5: SEM of coconut coir with 100x and 500x magnification

4.2.4.2. Coconut Coir Activated Carbon

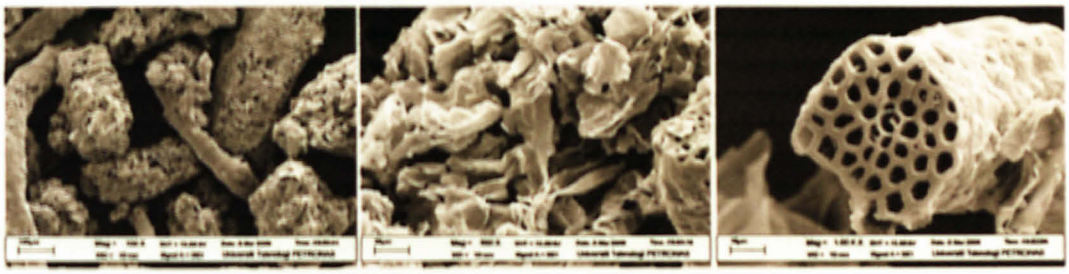


Figure 4.6: SEM of coconut coir activated carbon with 100x, 500x and 1000x magnification

4.2.4.3. Charcoal Activated Carbon

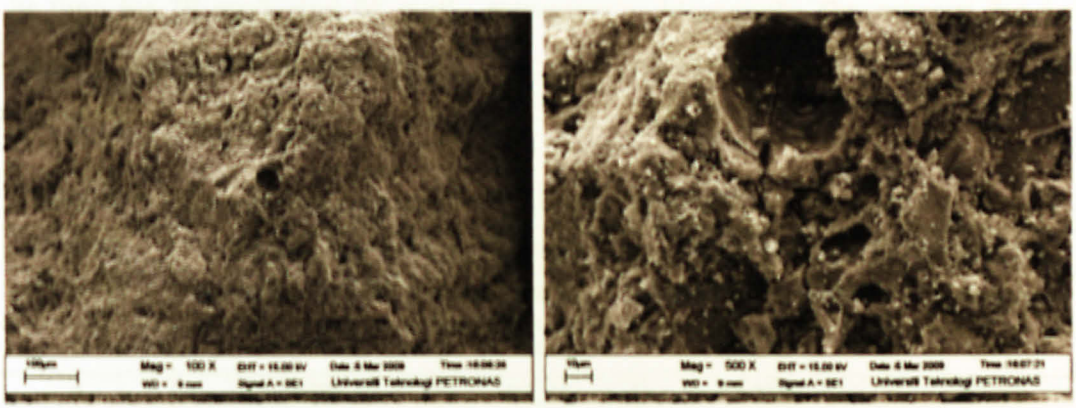


Figure 4.7: SEM of charcoal activated carbon with 100x and 500x magnification

4.3. Adsorption Study

4.3.1. Batch Study Using Coconut Coir Carbon

The adsorption rates of Acid Orange 7 and Acid Red dyes onto coconut coir carbon in a batch adsorption system by varying the parameters have been studied in this final year project II. The influence of experimental variables, such as contact time, solution concentration and carbon dose were evaluated. Meanwhile, the percentage of the adsorbate uptake can be calculated by following equation:

$$Adsorbate\ uptake\ (\%) = (C_o - C_t) / C_o \times 100$$

where C_o and C_t are the initial and at time (t) of the adsorbent concentration, respectively.

4.3.1.1. Effect of Initial Concentration and Contact Time

The effect of initial dye concentration and contact time on the removal of Acid Red and Acid Orange 7 is shown in the Table 4.4 and Table 4.5 below to investigate the efficiency of the coconut coir activated carbon. The contact time was varies every 1 hour time interval from 1 hour to 5 hours for both 20 mg/L and 40 mg/L solution concentration in a orbital shaker. Removal of Acid Red was found to be nearly 100%, at 2 hr time for 20 mg/L and 40 mg/L, respectively. Meanwhile, the removal for Acid Orange 7 was found to be nearly 100%, at 1 hr time for 20 mg/L and 40 mg/L, respectively.

Table 4.4: Effect of initial concentration and contact time on removal of Acid Red

Contact Time, hrs	Initial concentration= 20mg/L		Initial concentration = 40 mg/L	
	Absorbance (Abs)	% Removal	Absorbance (Abs)	% Removal
0	0.727	0.00	1.449	0.00
1	0.058	92.02	0.289	80.06
2	0.020	97.25	0.043	97.03
3	0.020	97.25	0.026	98.21
4	0.018	97.52	0.024	98.34
5	0.016	97.80	0.023	98.41

Table 4.5: Effect of initial concentration and contact time on removal of Acid Orange 7

Contact Time, hrs	Initial concentration= 20mg/L		Initial concentration = 40 mg/L	
	Absorbance (Abs)	% Removal	Absorbance (Abs)	% Removal
0	2.236	0.00	3.173	0.00
1	0.043	98.08	0.078	97.54
2	0.036	98.39	0.026	99.18
3	0.031	98.61	0.016	99.50
4	0.030	98.66	0.014	99.56
5	0.026	98.84	0.012	99.62

From the Figure 4.8 and Figure 4.9 below, the effect of initial dye concentration and contact time on the removal of Acid Red and Acid Orange 7 is shown. From the figure, the removal of dye increased with decreasing dye concentration for each dyes. The percentage removal of acid dye decreases as the concentration of the acid dye increases. Meanwhile the adsorption increased with an increase in contact time. The rate uptakes of removal for the first two hour is very fast and reach the equilibrium at 2 hr for Acid Red and 1 hr for Acid Orange 7.

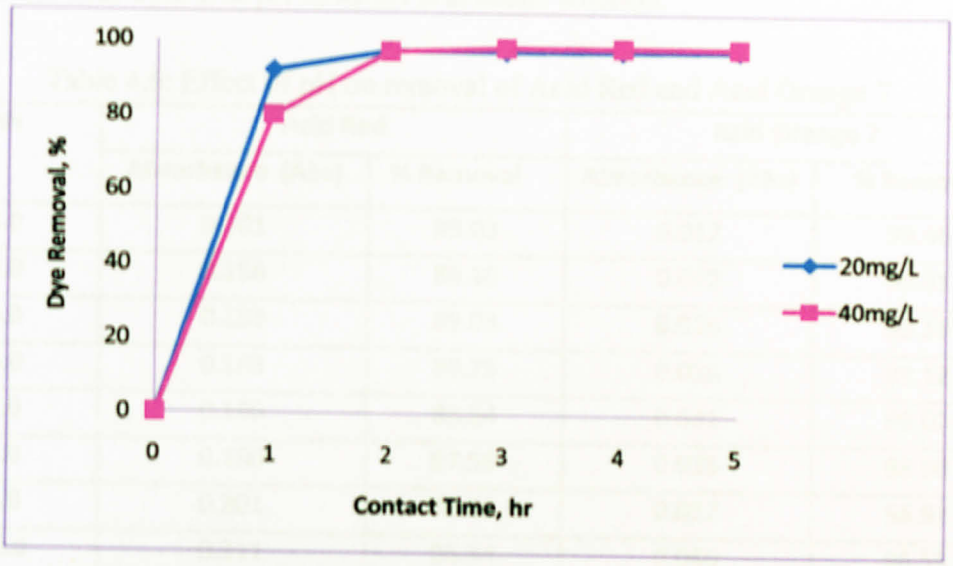


Figure 4.8: Effect of initial concentration and contact time on removal of Acid Red

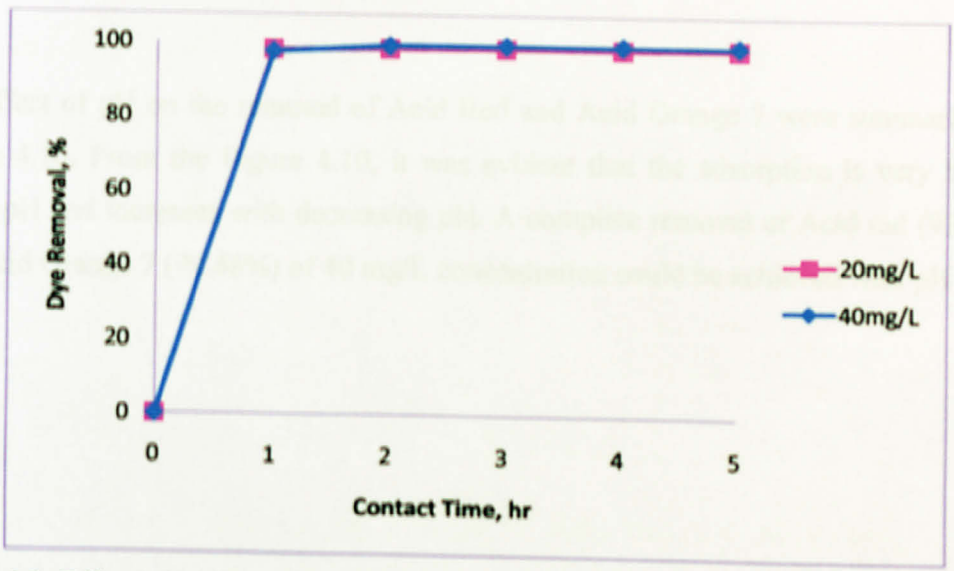


Figure 4.9: Effect of initial concentration and contact time on removal of Acid Orange7

4.3.1.2. Effect of pH

To determine the influence of pH, 100 mL of 40 mg/L solutions were added with 0.5 g of coconut coir activated carbon and the contact time used was 2 hours which the solution reached equilibrium. The pH of the solution was varied from pH 3 to pH 11. From the observation of the Table 4.6 of the effect of pH on the removal acid dyes, the dye uptake decreased with increasing pH of the dyes solution. The optimum value of dye removal for both dyes is at pH 3, which is at acidic solution.

Table 4.6: Effect of pH on removal of Acid Red and Acid Orange 7

pH	Acid Red		Acid Orange 7	
	Absorbance (Abs)	% Removal	Absorbance (Abs)	% Removal
3.0	0.101	93.03	0.017	99.46
4.0	0.158	89.10	0.022	99.31
5.0	0.159	89.03	0.026	99.18
6.0	0.163	88.75	0.028	99.12
7.0	0.166	88.54	0.031	99.02
8.0	0.180	87.58	0.035	98.90
9.0	0.201	86.13	0.037	98.83
10.0	0.211	85.44	0.046	98.55
11.0	0.270	81.37	0.048	98.49

The effect of pH on the removal of Acid Red and Acid Orange 7 were summarized in Figure 4.10. From the Figure 4.10, it was evident that the adsorption is very high at acidic pH and increases with decreasing pH. A complete removal of Acid red (93.03%) and Acid Orange 7 (99.46%) of 40 mg/L concentration could be achieved with pH 3.

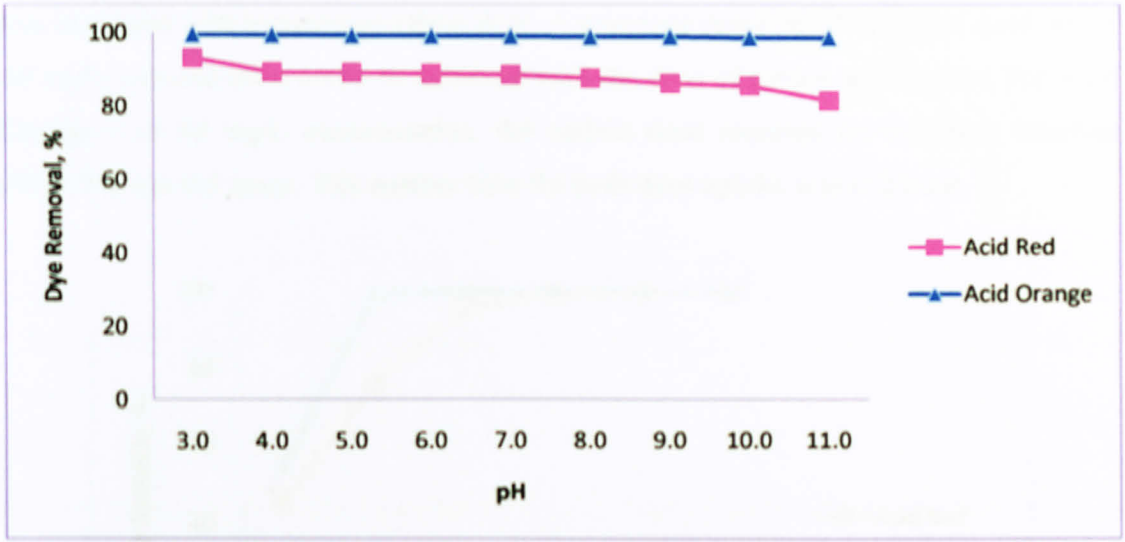


Figure 4.10: Effect of pH on removal of Acid Red and Acid Orange 7

4.3.1.3. Effect of Carbon Dose

From the observation of the Table 4.7 of the effect of carbon dosage on the removal acid dyes, the dye uptake increased with increasing dose of the carbon. To determine the influence of carbon dose, 100 mL of 40 mg/L solutions at pH 3 were added with different amount of coconut coir activated carbon varies from 0.2 gram to 1.0 gram. The contact time used was 2 hours.

Table 4.7: Effect of coconut coir carbon dosage on removal of Acid Red and Acid Orange 7

Carbon Dose, gram	Percentage Removal, %	
	Acid Red	Acid Orange 7
0.2	46.03	55.09
0.4	75.43	98.74
0.5	95.17	99.43
0.6	98.27	99.50
0.8	99.03	99.53
1.0	99.17	99.62

The effect of carbon dose on the removal of Acid Red and Acid Orange 7 were summarized in figure below. From the Figure 4.11, it was evident that the removal of

dye increased with increasing carbon dose. A complete removal (99.17%) of Acid red of 40 mg/L concentration could be achieved with the dose of carbon at 0.6 gram. For Acid Orange 7 of 40 mg/L concentration, the carbon dose required for complete removal (99.62%) was 0.5 gram. The contact time for both dyes uptake was at 2 hour.

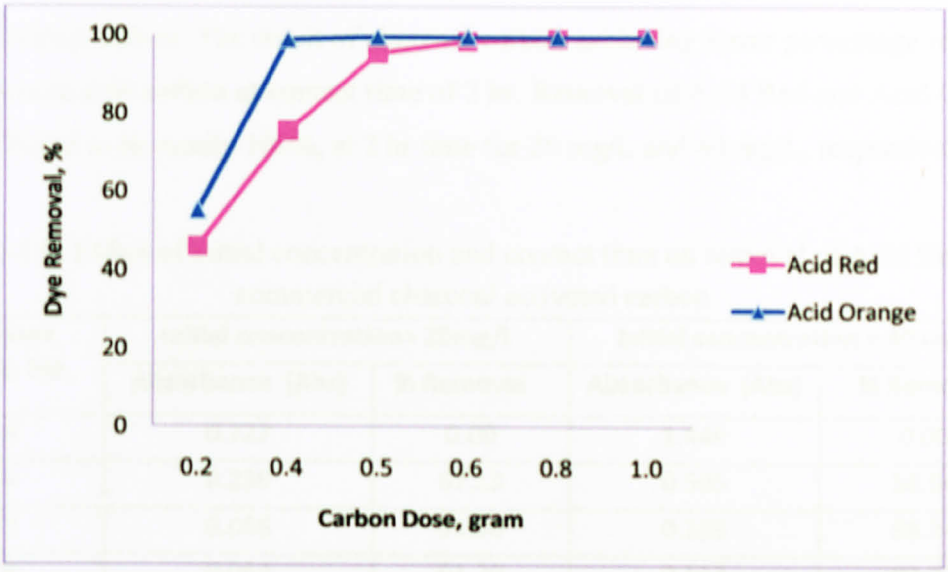


Figure 4.11: Effect of carbon dosage on removal of Acid Red and Acid Orange 7

4.3.2. Batch Study Using Commercial Charcoal Activated Carbon

A small amount of charcoal can clean a lot of waters based on the facts that have been studied by other researchers (Drake, 1994). The efficiency of coconut coir activated carbon on the adsorption process of color removal was compared to the commercial charcoal carbon. The adsorption rates of Acid Orange 7 and Acid Red dyes onto charcoal carbon in a batch adsorption system by investigating the effect of initial concentration and contact time on the acid dyes removal. The percentage of the adsorbate uptake can be calculated by the same method mention before:

$$\textit{Adsorbate uptake (\%)} = (C_o - C_t) / C_o \times 100$$

where C_o and C_t are the initial and at time (t) of the adsorbent concentration, respectively.

4.3.2.1. Effect of Initial Concentration and Contact Time

The effect of initial dye concentration and contact time on the removal of Acid Orange 7 and Acid Red by commercial charcoal carbon is shown in the tables and figures below. The procedure is about the same but the result obtained is totally different from coconut coir activated carbon. The result of charcoal carbon achieving lower percentage removal than coconut coir carbon at contact time of 2 hr. Removal of Acid Red and Acid Orange 7 were found to be nearly 100%, at 2 hr time for 20 mg/L and 40 mg/L, respectively.

Table 4.8: Effect of initial concentration and contact time on removal of Acid Red by commercial charcoal activated carbon

Contact Time, hrs	Initial concentration= 20mg/L		Initial concentration = 40 mg/L	
	Absorbance (Abs)	% Removal	Absorbance (Abs)	% Removal
0	0.727	0.00	1.449	0.00
1	0.239	67.13	0.595	58.94
2	0.065	91.06	0.169	88.34
3	0.064	91.20	0.112	92.27
4	0.050	93.12	0.108	92.55
5	0.048	93.40	0.080	94.48

Table 4.9: Effect of initial concentration and contact time on removal of Acid Orange 7 by commercial charcoal carbon

Contact Time, hrs	Initial concentration= 20mg/L		Initial concentration = 40 mg/L	
	Absorbance (Abs)	% Removal	Absorbance (Abs)	% Removal
0	2.236	0.00	3.173	0.00
1	0.339	84.84	0.758	76.11
2	0.054	97.58	0.064	97.98
3	0.054	97.58	0.064	97.98
4	0.053	97.63	0.050	98.42
5	0.052	97.67	0.040	98.74

From the Figure 4.12 and Figure 4.13, the rate uptake for the first two hours is very fast. The rate uptake is decreasing when the contact time increase until reached nearly 100% removal.

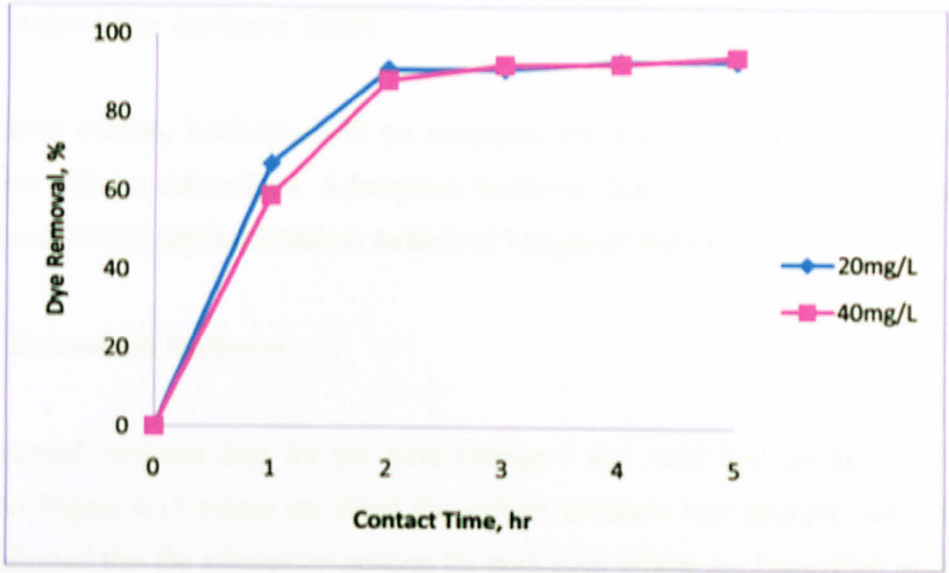


Figure 4.12: Effect of initial concentration and contact time on removal of Acid Red by commercial charcoal carbon

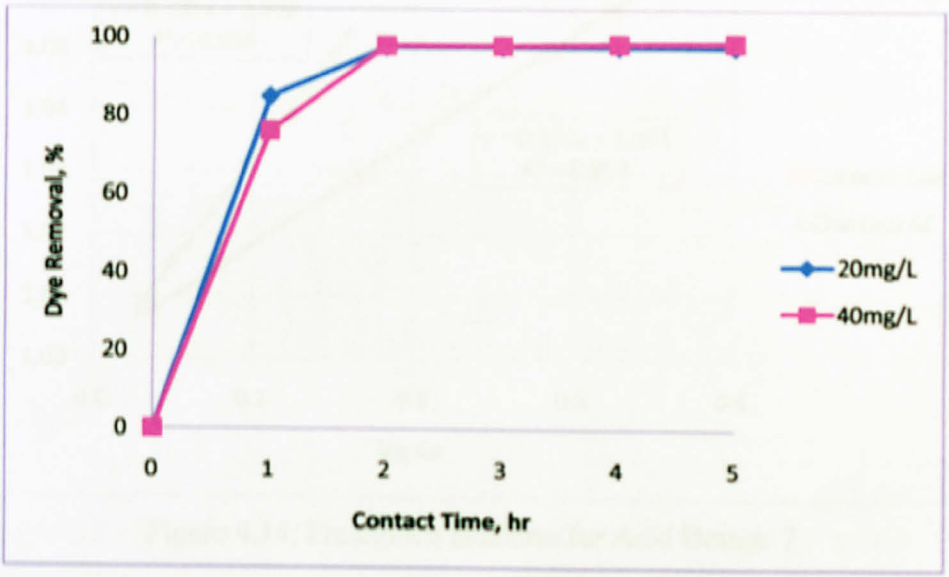


Figure 4.13: Effect of initial concentration and contact time on removal of Acid Orange7 by commercial charcoal carbon

The batch studies using aqueous solution were conducted after the coconut coir activated carbon was obtained. Only when the result obtained from the batch studies was favorable, the author will proceed with the Isotherms studies.

4.4. Adsorption Isotherm Study

In the next studies, Isotherms will be measured and the isotherm parameters for the isotherms will be determined. Adsorption isotherm data of dyes were fitted to well-known and widely applied isotherm models of Langmuir and Freundlich.

4.4.1. Freundlich Isotherm

Experimental isotherm data for the Acid Orange 7 and Acid Red are given in Figure 4.14 and Figure 4.15 where the fitted Freundlich isotherm best straight line is shown. This indicated that the adsorption process for both dyes follow the Freundlich model.

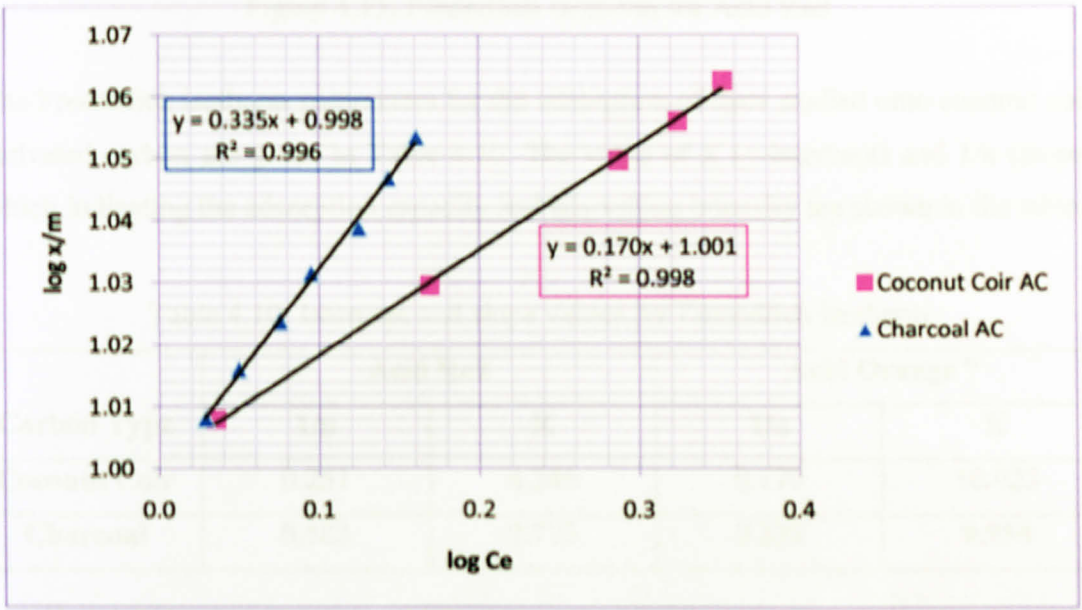


Figure 4.14: Freundlich isotherm for Acid Orange 7

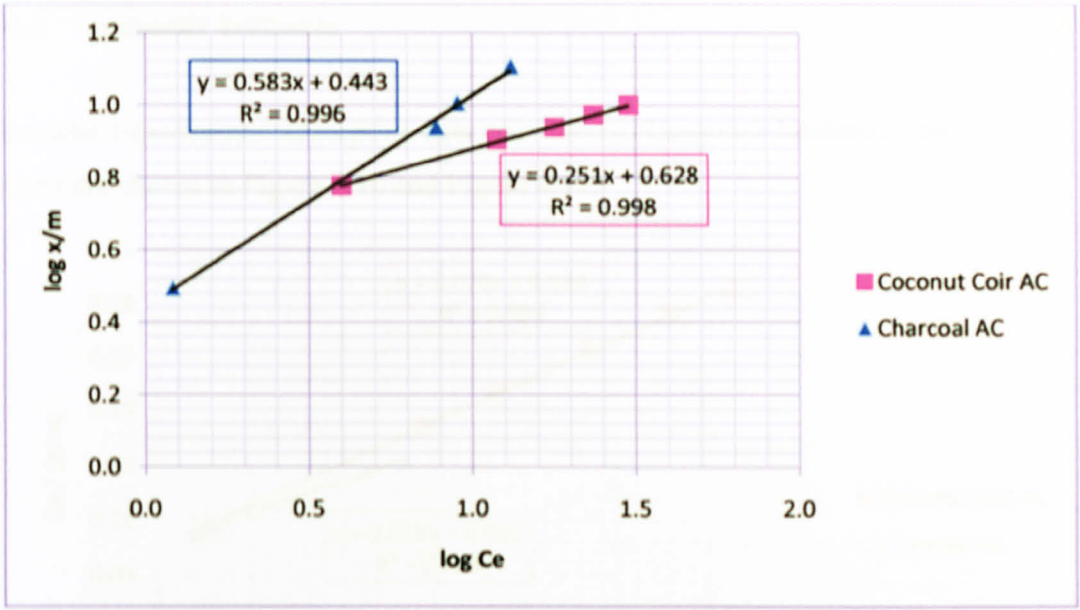


Figure 4.15: Freundlich isotherm for Acid Red

The Freundlich isotherm parameters for the adsorption of dyes studied onto coconut coir activated carbon are given in Table 4.10. The value of *K* (y-intercept) and *1/n* (slope) which indicating the adsorption capacity and adsorption intensity are shown in the table.

Table 4.10: Intercept and slope values for Freundlich isotherm

Carbon Type	Acid Red		Acid Orange 7	
	1/n	K	1/n	K
Coconut Coir	0.251	4.246	0.170	10.023
Charcoal	0.583	2.773	0.335	9.954

Conducting adsorption isotherm studies has shown that the coconut coir activated carbon has higher adsorption capacity compared to commercially charcoal activated carbon. The adsorption intensity of charcoal activated carbon is higher for both Acid Red and Acid Orange 7. This proven that the coconut coir activated carbon prepared which is low cost is much better compared to the commercial activated carbon which is very highly in production cost.

4.4.2. Langmuir Isotherm

The same experimental adsorption data was fitted to Langmuir Isotherm. The plotted graphs are shown in Figure 4.16 and Figure 4.17.

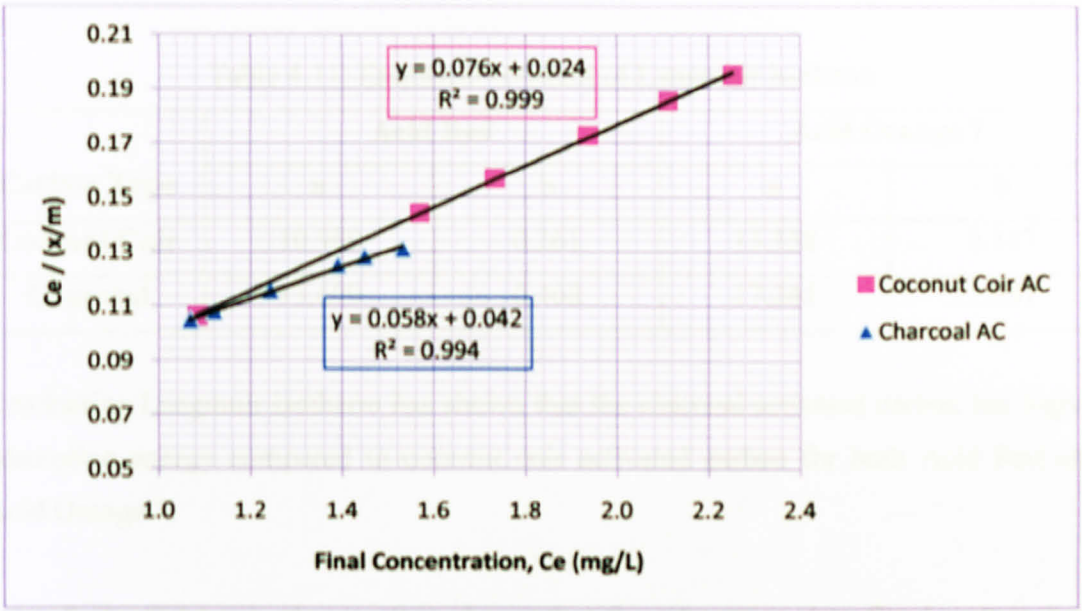


Figure 4.16: Langmuir isotherm for Acid Orange 7

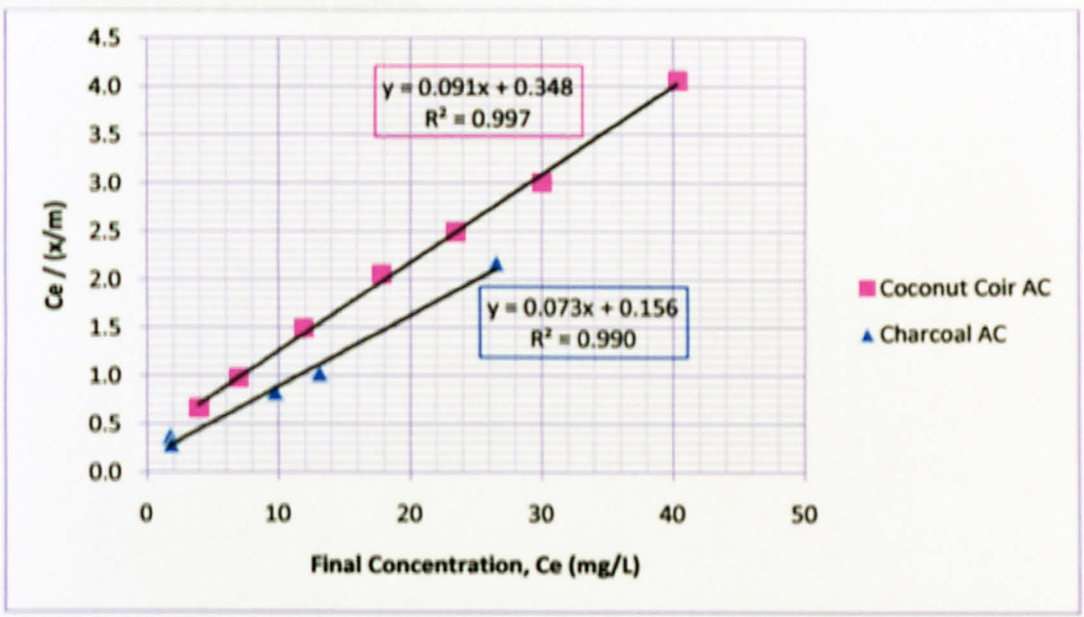


Figure 4.17: Langmuir isotherm for Acid Red

Adsorption capacities and adsorption energy of both coconut coir activated carbon and commercial charcoal activated carbon for the dyes determined by empirical constant *a* and *b* of Langmuir model. The Empirical constants are summarized in Table 4.11 by determining the slope and y-intercept value of the best-fit line.

Table 4.11: Empirical constant of Langmuir isotherm

Carbon Type	Acid Red		Acid Orange 7	
	a	b	a	b
Coconut Coir	10.989	0.261	13.158	3.167
Charcoal	13.699	0.468	17.241	1.381

Conducting Langmuir isotherm has shown that the charcoal activated carbon has higher adsorption energy compared to coconut coir activated carbon for both Acid Red and Acid Orange 7.

From both of these isotherms, it is shown that the adsorption data fitted well to both Freundlich and Langmuir isotherm for the adsorption of Acid Orange 7 and Acid Red onto coconut coir activated carbon.

CHAPTER 5

CONCLUSION

The results of this study showed that the activated carbon prepared from coconut coir has suitable adsorption capacity for the removal of Acid Orange 7 and Acid Red from aqueous solution. Adsorption increased with increase in contact time and carbon dose and decreased with increase in dye solution concentration at the optimum pH 3. According to the Freundlich isotherm, coconut coir activated carbon has higher adsorption capacity and lower adsorption intensity for the acid dyes compared to the charcoal activated carbon. According to Langmuir isotherm, the coconut coir activated carbon has appreciable adsorption capacity but lower adsorption energy for the acid dyes compared to the charcoal activated carbon.

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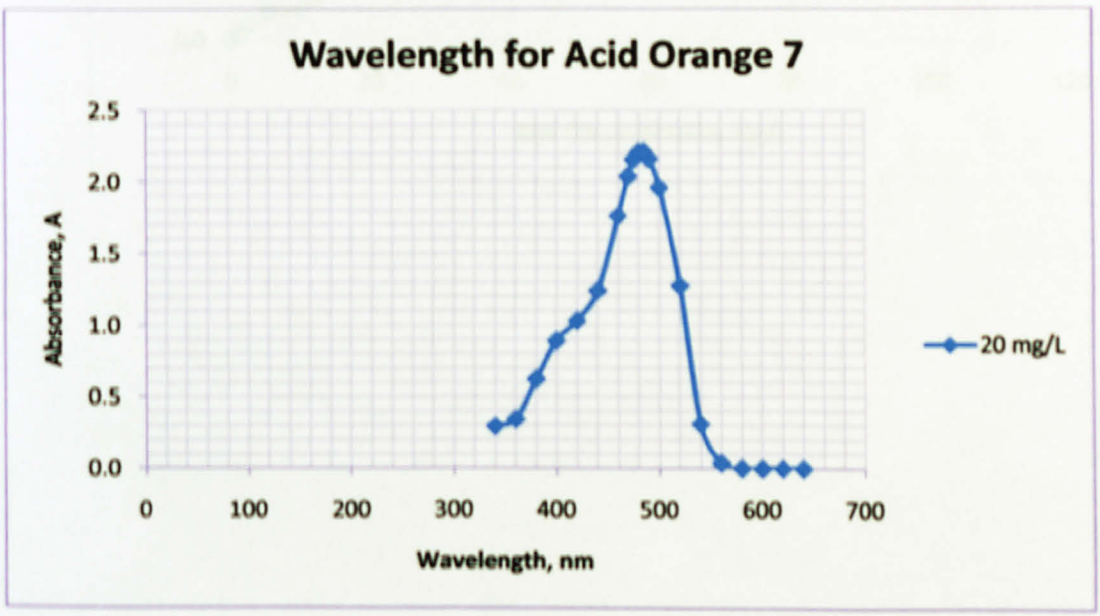
Appendix A: Data of Acid Orange 7

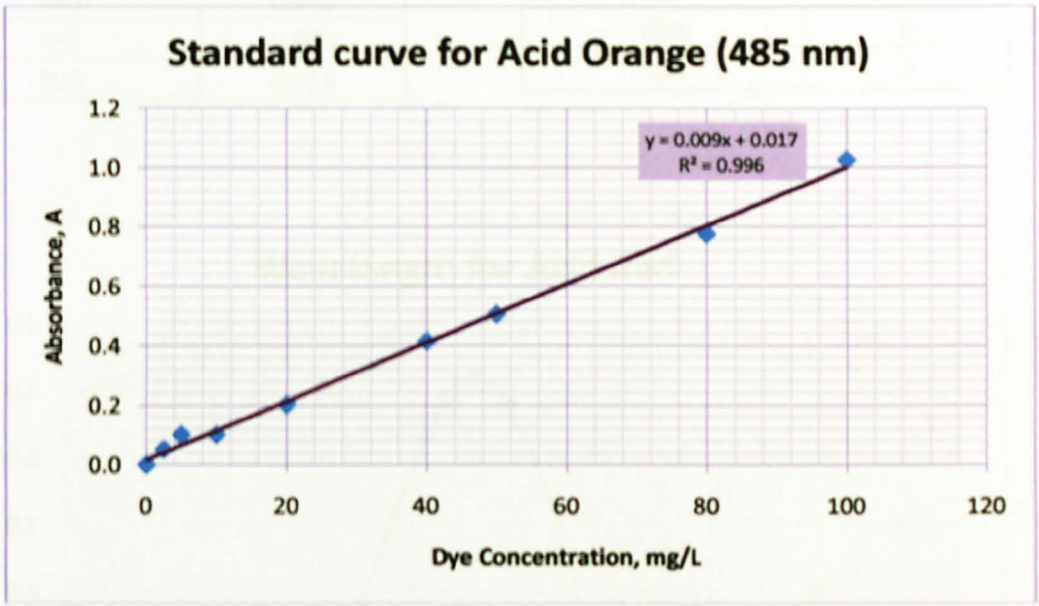
Standard wavelength of maximum absorbance (concentration=20 mg/L):

Wavelength, nm	Absorbance, A
340	0.295
360	0.342
380	0.622
400	0.890
420	1.029
440	1.240
460	1.764
470	2.037
475	2.156
480	2.209
485	2.210
490	2.162
500	1.960
520	1.277
540	0.310
560	0.039
580	0.003
600	0.000
620	0.000
640	0.000



Standard Curve for Acid Orange (Wavelength = 485 nm)

concentration	absorbance
0	0.000
2.5	0.052
5	0.102
10	0.102
20	0.203
40	0.415
50	0.506
80	0.777
100	1.026

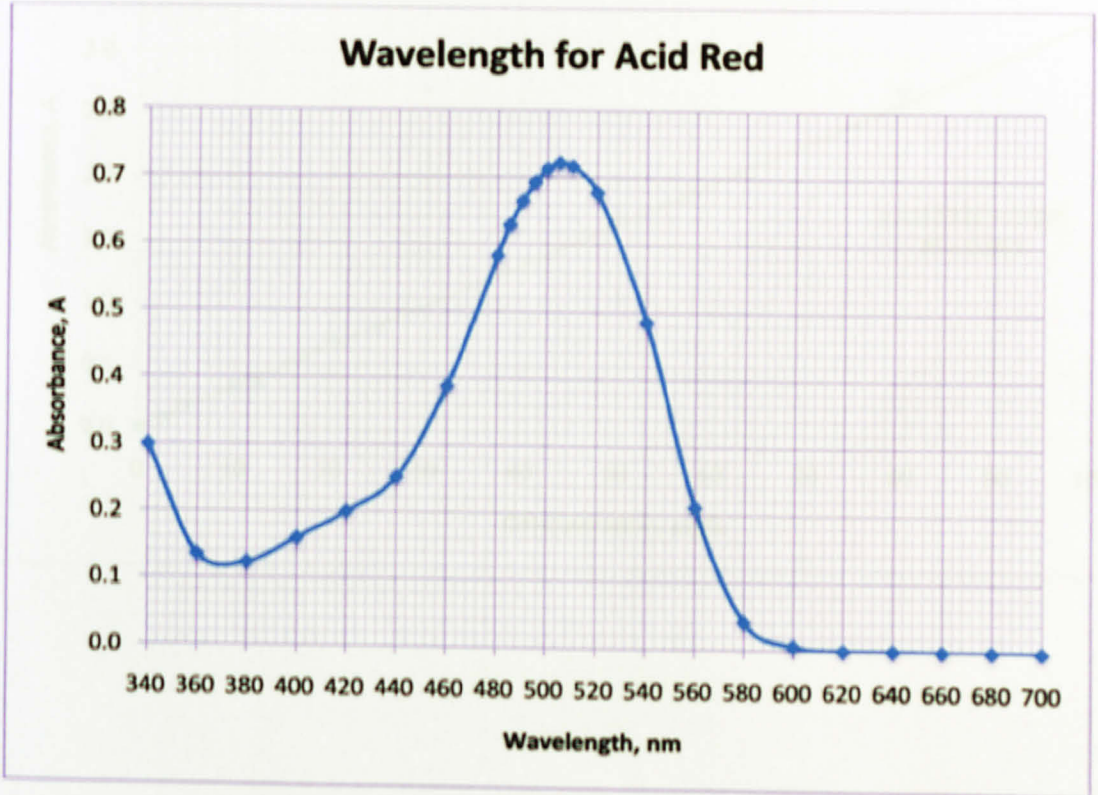


Appendix B: Data of Acid Red

Standard wavelength of maximum absorbance (concentration=20 mg/L):

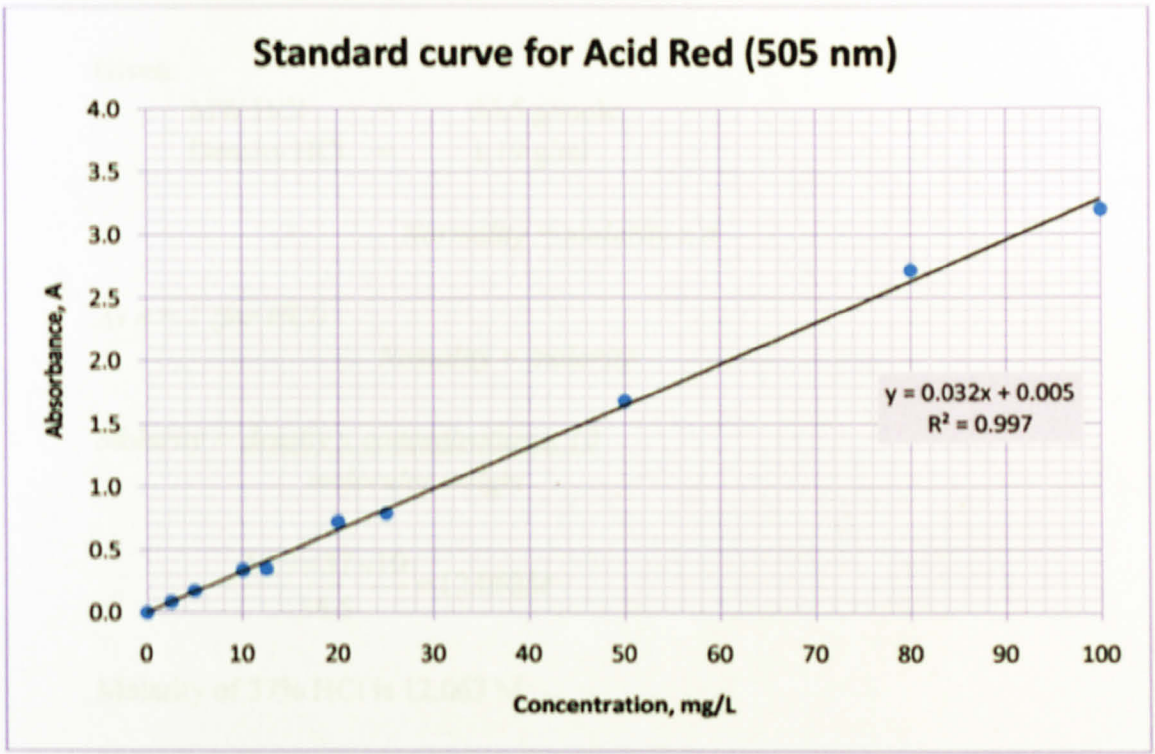
Wavelength, nm	Absorbance, A
340	0.299
360	0.135
380	0.123
400	0.16
420	0.2
440	0.252
460	0.388
480	0.583
485	0.629
490	0.665
495	0.693
500	0.713

Wavelength, nm	Absorbance, A
505	0.722
510	0.717
520	0.678
540	0.486
560	0.211
580	0.042
600	0.007
620	0.001
640	0.001
660	0
680	0
700	0



Standard Curve for Acid Red (Wavelength = 505 nm)

Concentration mg/L	Absorbance, A
0	0
2.5	0.082
5	0.175
10	0.336
12.5	0.347
20	0.722
25	0.791
50	1.681
80	2.717
100	3.197



Appendix C: Solution Preparation for Acid

- **For 10% HCl with initial concentration 37%**

$$M_1V_1 = M_2V_2$$

$$V_1 = \frac{(10\%)(1000ml)}{37\%}$$

$$V_1 = 270.270ml$$

- 270.270 ml of HCl with initial concentration 37% is needed to be diluted with distilled water to produce 10% HCl

- ***For 0.02 N HCl with initial concentration 37%***

Given:

MW HCl = 36.5 g/mole
Density HCl = 1.19 g/ml

Normality = molarity x *n*

As n = 1 (for HCl)

Nomality = molarity

$$Molarity = \frac{density \times concentration \times 10}{molecular\ weight}$$

$$= \frac{1.19 \times 37 \times 10}{36.5} = 12.063M$$

Molarity of 37% HCl is 12.063 M

Thus, to calculate 0.02 M = 0.02 N

$$V_1 = \frac{(0.02M)(1000ml)}{12.063M}$$

$$V_1 = 1.658ml$$

Appendix D: Adsorption Isotherm of Acid Orange 7

COCONUT COIR ACTIVATED CARBON

C_o	Abs_o	Abs_e	C_e	C_o-C_e	$(C_o-C_e)/C_o$	V	$(C_o-C_e)V$	m	x/m	$C_e/(x/m)$	log C_e	log (x/m)
51	3.306	0.060	0.92559	50.07441	0.98185	0.1	5.00744	0.5	10.01488	0.09242	-0.03358	1.00065
52	3.308	0.069	1.08464	50.91536	0.97914	0.1	5.09154	0.5	10.18307	0.10651	0.03529	1.00788
53	3.310	0.080	1.28097	51.71903	0.97583	0.1	5.17190	0.5	10.34381	0.12384	0.10754	1.01468
54	3.313	0.085	1.38545	52.61455	0.97434	0.1	5.26145	0.5	10.52291	0.13166	0.14159	1.02214
55	3.317	0.089	1.47573	53.52427	0.97317	0.1	5.35243	0.5	10.70485	0.13786	0.16901	1.02958
56	3.319	0.093	1.56915	54.43085	0.97198	0.1	5.44309	0.5	10.88617	0.14414	0.19566	1.03688
57	3.321	0.101	1.73351	55.26649	0.96959	0.1	5.52665	0.5	11.05330	0.15683	0.23893	1.04349
58	3.324	0.111	1.93682	56.06318	0.96661	0.1	5.60632	0.5	11.21264	0.17274	0.28709	1.04971
59	3.327	0.119	2.11031	56.88969	0.96423	0.1	5.68897	0.5	11.37794	0.18547	0.32435	1.05606
60	3.330	0.125	2.25225	57.74775	0.96246	0.1	5.77477	0.5	11.54955	0.19501	0.35262	1.06257

CHARCOAL ACTIVATED CARBON

C_o	Abs_o	Abs_e	C_e	C_o-C_e	$(C_o-C_e)/C_o$	V	$(C_o-C_e)V$	m	x/m	$C_e/(x/m)$	log C_e	log (x/m)
51	3.306	0.062	0.95644	50.04356	0.98125	0.1	5.00436	0.5	10.00871	0.09556	-0.01934	1.00038
52	3.308	0.068	1.06892	50.93108	0.97944	0.1	5.09311	0.5	10.18622	0.10494	0.02895	1.00801
53	3.310	0.070	1.12085	51.87915	0.97885	0.1	5.18792	0.5	10.37583	0.10802	0.04955	1.01602
54	3.313	0.073	1.18986	52.81014	0.97797	0.1	5.28101	0.5	10.56203	0.11265	0.07550	1.02375
55	3.317	0.075	1.24359	53.75641	0.97739	0.1	5.37564	0.5	10.75128	0.11567	0.09468	1.03146
56	3.319	0.079	1.33293	54.66707	0.97620	0.1	5.46671	0.5	10.93341	0.12191	0.12481	1.03876
57	3.321	0.081	1.39024	55.60976	0.97561	0.1	5.56098	0.5	11.12195	0.12500	0.14309	1.04618
58	3.324	0.083	1.44826	56.55174	0.97503	0.1	5.65517	0.5	11.31035	0.12805	0.16085	1.05348
59	3.327	0.084	1.48963	57.51037	0.97475	0.1	5.75104	0.5	11.50207	0.12951	0.17308	1.06078
60	3.330	0.085	1.53153	58.46847	0.97447	0.1	5.84685	0.5	11.69369	0.13097	0.18513	1.06795

Appendix E: Adsorption Isotherm of Acid Red

COCONUT COIR ACTIVATED CARBON

C_o	Abs_o	Abs_e	C_e	C_o-C_e	$(C_o-C_e)/C_o$	V	$(C_o-C_e)V$	m	x/m	$C_e/(x/m)$	log C_e	log (x/m)
10	0.373	0.001	0.02681	9.97319	0.99732	0.1	0.99732	0.6	1.66220	0.01613	-1.57171	0.22068
20	0.728	0.012	0.32967	19.67033	0.98352	0.1	1.96703	0.6	3.27839	0.10056	-0.48192	0.51566
30	1.045	0.039	1.11962	28.88038	0.96268	0.1	2.88804	0.6	4.81340	0.23260	0.04907	0.68245
40	1.447	0.144	3.98065	36.01935	0.90048	0.1	3.60194	0.6	6.00323	0.66309	0.59995	0.77838
50	1.776	0.248	6.98198	43.01802	0.86036	0.1	4.30180	0.6	7.16967	0.97382	0.84398	0.85550
60	2.127	0.422	11.90409	48.09591	0.80160	0.1	4.80959	0.6	8.01598	1.48504	1.07570	0.90396
70	2.415	0.616	17.85507	52.14493	0.74493	0.1	5.21449	0.6	8.69082	2.05447	1.25176	0.93906
80	2.774	0.814	23.47513	56.52487	0.70656	0.1	5.65249	0.6	9.42081	2.49184	1.37061	0.97409
90	3.040	1.014	30.01974	59.98026	0.66645	0.1	5.99803	0.6	9.99671	3.00296	1.47741	0.99986
100	3.248	1.310	40.33251	59.66749	0.59667	0.1	5.96675	0.6	9.94458	4.05573	1.60566	0.99759

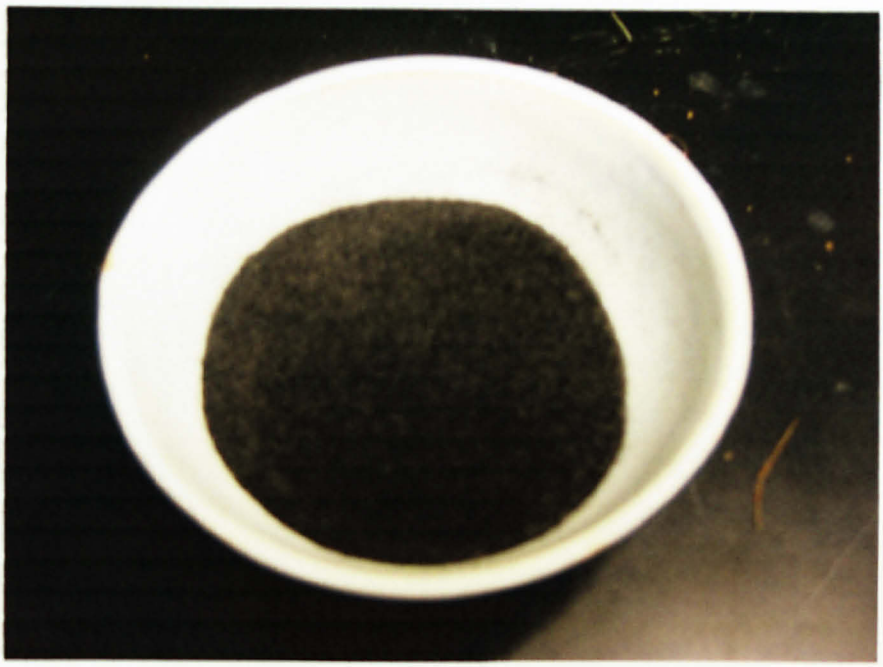
CHARCOAL ACTIVATED CARBON

C_o	Abs_o	Abs_e	C_e	C_o-C_e	$(C_o-C_e)/C_o$	V	$(C_o-C_e)V$	m	x/m	$C_e/(x/m)$	log C_e	log (x/m)
10	0.373	0.034	0.91153	9.08847	0.90885	0.1	0.90885	0.6	1.51475	0.60177	-0.04023	0.18034
20	0.728	0.044	1.20879	18.79121	0.93956	0.1	1.87912	0.6	3.13187	0.38596	0.08235	0.49580
30	1.045	0.061	1.75120	28.24880	0.94163	0.1	2.82488	0.6	4.70813	0.37195	0.24333	0.67285
40	1.447	0.066	1.82446	38.17554	0.95439	0.1	3.81755	0.6	6.36259	0.28675	0.26114	0.80363
50	1.776	0.234	6.58784	43.41216	0.86824	0.1	4.34122	0.6	7.23536	0.91051	0.81874	0.85946
60	2.127	0.275	7.75740	52.24260	0.87071	0.1	5.22426	0.6	8.70710	0.89093	0.88972	0.93987
70	2.415	0.311	9.01449	60.98551	0.87122	0.1	6.09855	0.6	10.16425	0.88688	0.95494	1.00708
80	2.774	0.338	9.74766	70.25234	0.87815	0.1	7.02523	0.6	11.70872	0.83251	0.98890	1.06851
90	3.040	0.443	13.11513	76.88487	0.85428	0.1	7.68849	0.6	12.81414	1.02349	1.11777	1.10769
100	3.248	0.863	26.57020	73.42980	0.73430	0.1	7.34298	0.6	12.23830	2.17107	1.42439	1.08772

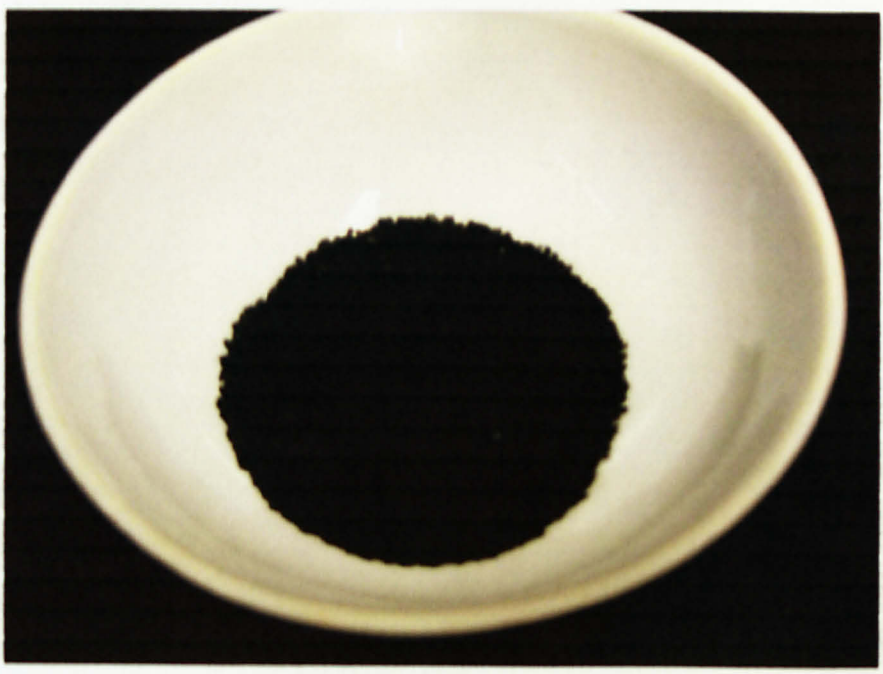
Appendix F: Sample of coconut coir, coconut coir activated carbon and charcoal activated carbon used for batch study and adsorption study

Coconut

Coconut coir



Coconut coir activated carbon



Charcoal activated carbon